The use of control charts for the quality control of analytical determinations in soap and detergents.

By

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01/AS/050

This thesis is submitted in partial fulfillment of the report for the degree of

Bachelor of science

In

Physical sciences.

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Buttala.

July 2005

DECLARATION

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

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ACKNOWLEDGEMENT

I wish to express my deepest apprection and gratitude to Dr. D. B. M. Wickramarathna, Dean, Faculty of Applied Sciences and Dr. Nirmali Wickramarathna (internal Supervisor), Head, Department of Physical Sciences , for giving this greate Opportunity to train in a highly competitive industrial environment in a reputed firm ,Uniliver Ceylon Limited and for their supervision , valuable guidance and encouragement .

I am grateful to my external supervisor Dr. Ruwan Pathirana, Laboratory manager, Uniliver Ceylon Limited, for his supervisor and guidance.

I would like to thank Mr. Pani Ardehetti, Mr. Chaminda Munasingha, Mr. Ruchira Perera, Mr. Waruna, Mr. Mohan, Mr. Thissa Atthanayaka, Mr. Upali Senawirattna, Mr. Mewan Perera, Mrs. Gayatthri and all other laboratory staff for their valuable support during training period.

P.M.W. Jayasekara

ABSTRACT

The accuracy of an analytical determination is very important in an industrial processes .The quality of analytical determinations can be performed a Shewhart control chart. The control chart provides interpretation of standard of a a analytical determination can be identified in control or out of control.

A suitable check sample was selected to construct a control chart .This check sample need not be identical in old aspect to the sample was sufficiently similar to the test sample in chemical composition and physical state (solid, liquid, or gas) so that the conclusion drawn from the control chart are relevant to the subject subsequent analyses.

After selecting a check sample, start the experiments with the standard procedure . Under these condition composed a training set of 20 initial, independent observation for the check sample .Determine the average and the standard deviation of the training set . Enter all 20 observation in a control chart .Then continue the standard procedure for once a day. Once the training set has been completed the control chart is ready to accept new observations from subsequent determinations of the check sample. New data entered in the control chart reveal whether or not the analytical determination is in control. For this decision we use the warning limits (2s) and the control limits (3s) from their definition it can statistically be expected that one out of every 20 observations will exceed the warning limits and one out of every thousand the control limits.

In the training period, for selected analytical determinations were used to check the in control or out of control .There were no any out of result in selected analytical determinations. All the analytical determinations were in control.

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1. Select suitable check samples for each analysis
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3. Evaluation of expiry dates for checks samples.
4. Implementation of control charts for ongoing analytical system.

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CHAPTER 01 INTRODUCTION

Uniliver Sri Lanka Limited is a largest company in Sri Lanka, which produced soaps, detergents, Foods and personal products. The soap and detergent manufacturing is very important of them. There are several very important testings and experiments are used in this manufacturing process.

1. Determination of Glycerol in crude Glycerine.
2. Determination of NaCl content in soap.
3. Determination of Total Fatty Matter by Ether extraction method.
4. Determination of Anaionic detergent in detergents.
5. Determination of moisture of soap and Detergent
6. Determination of titer value of soap
7. Determination of free alkali (caustic) in soap

Due to the impotence of the above experiments, the standardizing analytical determinations used in quality control determinations are very important. In the training period, some selected analytical determinations used to develop a overall chart to maintain the quality.

The quality of analytical determinations following a standard procedure can be controlled through the periodic analysis of a check sample. This method describes the use of the Shewhart control chart to judge whether the result of the check sample falls within the normal variability of the procedure or whether corrective action need to be taken.

CHAPTER 02 LITRETURE REVIEVE

2:1 Glycerol

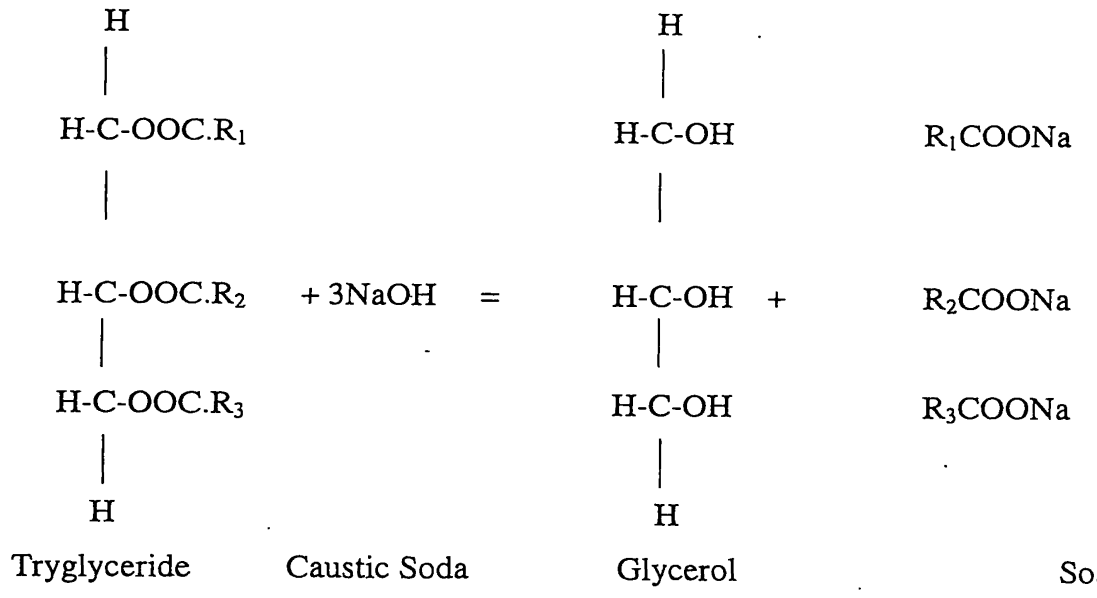
Glycerol (glycerine) was discovered in 1779 by the Swedish chemist Scheele. Commercially known as glycerine, it is a try-basic alcohol with a Conventional formula $-CH_2OH-CHOH-CH_2OH$. Glycerine has always been an expensive commodity in the world markets due to the high demand caused by its very wide range of use. Users are heavily dependent on the soap and fatty acid industry for the supply of a major part of their requirements. Most often the economics of the soap and fatty acid manufacturing industry is influenced by the credit available from the sale of glycerine as a by-product. However, the price is generally related to that of synthetic glycerine. Due to the over abundance of synthetic glycerine in the world, the price of refined glycerine –apart from short term fluctuations- remains buoyant to soap manufacturers.

As glycerine is used in foods, medical, personal care products, as well as for other purposes which require a high degree of purity, special attention is paid to the recovery and processing of glycerine. Manufacturing any substance in a state of high purity often depends upon the complexity of the compound and the mixture in which it exist. Modern technology has made it possible to produced refined glycerine from natural sources that are almost identical in quality to that of synthetic glycerine.

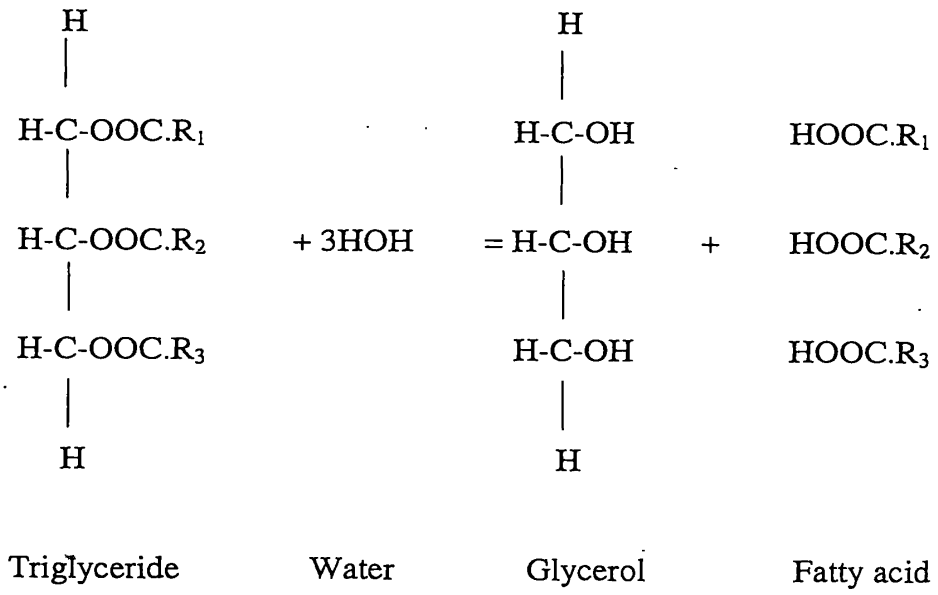
2:1:1 Method of production of Glycerol

Although a large proportion of glycerol is derived from the direct saponification of fats with caustic soda and recovery as a by-product from the resulting soap, an important part is made by either the hydrolyzing or the splitting of fats in to free fatty acids and glycerol.

During the saponification process, glycerol is liberated and then extracted by the counter current washing of soap to obtain spent lye that has a maximum concentration of glycerol in water. The saponification reaction is:



The hydrolysis of fats and oils is a reversible reaction, which takes place in stages and produces glycerol and fatty acids. Glycerol is removed from the system as an aqueous solution called sweet water.



From the point of view of glycerine production, hydrolysis has the advantage that the glycerol may be obtained directly, in a relatively concentrated solution.

Direct saponification forms soap, which has to be separated from the glycerol. Both spent lye and sweet water continue to be the major row materials necessary for manufacturing refined glycerine from natural sources.

2:1:2 Synthetic Glycerine

Different processes are available to synthetically produce glycerine. More modern methods derive their raw material from petroleum sources. Three routes, all using propylene as the basic raw material, have been used commercially.
The quality of synthetic glycerine that is currently being produced is very high. Refiners of natural glycerine often find it difficult to match the quality of the synthetic product. Improvements made in the refining of natural glycerine have produced a quality that approaches that of the synthetics.

2:1:3 Glycerine Specifications

Glycerine is produced and marketed in various grades and specifications, as listed and described as follows.

2:1:3:1 Crude Glycerine

Soap-lye crude glycerine, 80% produced by the manufacture of soap, and saponification crude glycerine, 88% produced by the manufacture of fatty acids, are important articles of commerce. But these are almost never used with out further refining.

2:1:3:2 Refined Glycerine

Refined glycerine of USP and CP, 99.5,99 and 96% grades are normally marketed meeting the specifications of the U.S. Pharmacopoeia standard specifications for chemically pure glycerine.

2:1:3:3 High Gravity Glycerine

High gravity glycerine (99%) is close to colorless, and conforms to federal specification 0.G-419b.

2:1:4 Refined Glycerine Market

The world production capacity of refined glycerine is estimated at 1.5 billion pounds, 86% of which is form natural resources. The United States accounts for about 450 million pounds. Dow is the only single source of synthetic glycerine in the United States. The pricing of natural glycerine is influenced by both the quantity and the price of synthetic glycerine. The current U.S. market is estimated to be about 340 million pounds. Drugs and personal care products account for about 40% of the demand, followed by tobacco (15%), and food industries (15%).

2:1:5 Theory

In the test for Glycerol,

$$\text{Glycerol} + NaIO_4 \rightarrow HCHO + HCOOH$$

Ethylene Glycol is added to remove excess $NaIO_4$. The formic acid formed is estimated by titrating with NaOH.

2:2 Sodium Chloride

Sodium Chloride is a very important factor in soap industry. Normal NaCl percentage of soap is about 0.7-0.8. If NaCl percentage is high, soap will be hard than the normal state. If the NaCl percentage is low, soap will be soft.

2:2:1 Theory:

$NaCl + AgNO_3 \rightarrow AgCl + NaNO3$

When Cl is over,

$K_2CrO_4 + 2AgNO3 \rightarrow Ag2CrO4 + 2KNO3$

2:3 Total Fatty Matter (TFM)

Total fatty matter is also very important in soap industry. This factor is used for identify the hard soaps and Toilet soaps. In Toilet soap, TFM is high. Normally, it is about 75-79 %. TFM of hard soap is low. It is about 63%.

2:3:1 Theory

In this method, the soap is dissolved in water and when 4N H_2SO_4 is added all fatty mater in soap is separated and extracted to an ether layer. The fatty material can be isolated by evaporating the ether and during in and oven.

2:4 Total Anionic Detergents

This method is applicable to the analysis of alkylbanzene sulphonates, alkylsulphonates, alkylsulphates, and hydroxy sulphates in detergent powder. The total active detergent is determined if a mixture of sulphonates and sulphates. This procedure will determine the total amount of anionic detergents. The mean molecular

weight of the active detergent must be known. Sulphonates with low molecular weight such as toluene and xylen sulphonates do not interfere when present in concentrations up to 15% of the active material.

2:4:1Theory:

An aqueous solution of the sample is titrated with a standard cationic active solution in a two-phase water-chloroform system using a mixture of a cationic dye (dimidum bromide) and cationic dye (disulphine blue v) as indicator. The anionic surfactant forms a salt with the cationic dye, which dissolves in the chloroform layer to give this layer a red pink color.

The end point is reached when the Hyamine cation displaces the dimidum cation from the chloroform soluble salt and the pink color leaves the chloroform layer as the dye passes to the aqueous phase. Hyamine added in excess forms a salt with the anionic dye disulphine blue v, which dissolves in the chloroform layer and colors it blue.

Due to the impotency of above experiments, the quality control of analytical determinations is very important.

The quality of an analytical determination following a standard procedure can be controlled through the periodic analysis of a check sample. This method describes the use of the Shewhart control chart to judge whether the result of the check sample falls within the normal variability of the procedure or whether corrective action need to be taken.

2: 5 Check Sample

The check sample need not be identical in all aspects to the sample 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 conclusion drawn from the control chart are relevant to the subsequent analysis. In particular the component that is the subject of a chemical analysis must be present in the check sample in the same form as in the daily samples.

The check sample must be stored under the 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 afresh result in the control chart. For a frequent analysis this could be daily, but no less than once a week.

CHAPTER 03 METHODOLOGY

3.1 Determination of Glycerol in crude Glycerine

Apparatus

Electric balance, Titration flask, Burret

Chemicals

Phenol red indicator, N/50 H_2SO_4, Dilute NaOH, Sodium Meta per iodate ($NaIO_4$), 50% Ethylene Glycol, N/10 NaOH, Distilled water

Check sample: Purified Glycerin

Procedure

About 0.2000 g – 0.2500 g of crude glycerine sample was weighted. A few drops of phenol red indicator were added. (If alkaline, neutralize with dilute acid and make just alkaline with dilute NaOH) 50 ml of $NaIO_4$ was added. It was kept in a dark place for 60 minutes. 10 ml of 50% Ethylene Glycol was added. Then it was kept in dark place for 30 minutes. The solution was titrated with N/10 NaOH solution. A blank determination was carried out using 200 ml of water.

3.2 Determination of NaCl in soap

Apparatus

Beaker, Electric balance, Water bath, Volumetric flask, Wash bottle, Filter paper, Measuring cylinder

Chemicals

20% Calcium Nitrate - $Ca(NO_3)_2$, Phenolphthalein, N/10 $AgNO_3$, Cromate indicator, Distilled water

Check sample: Potassium Chloride

Procedure

For soaps

Ten grams of soap was weighed in to a well-washed beaker. It was dissolve in water by boiling. 25 ml of 20% $Ca(NO_3)_2$ was added. It was swirled until the precipitation is completed. Then, it was filtered through a filter paper in to a 250ml volumetric flask with the help of wash bottle and it was made up to the mark. 25 ml of the filtrate was pipeted out and a drop of phenolphthalein indicator was added. (If it is alkaline, neutralize with N/50 HNO_3 or N/50 H_2SO_4). Then, it was titrated with N/10 $AgNO_3$ solution using Chromate indicator.

For check sample

About 0.1000 g of sample was weighed. It was dissolve in distilled water. A drop of phenolphthalein indicator. Then, it was titrated with N/10 $AgNO_3$ solution using Chromate indicator.

3. 3 Determination of Total Fatty Matter by Ether extraction method.

Apparatus

Beaker, Electric balance, 500 ml Separating funnel, Water bath, Stopper, Wash bottle, Round bottom flask, Soxhelt

Chemicals

4N H_2SO_4, Diethyl ether, Methyl orange indicator, Acetone

Check sample: Base soap

Procedure

Ten grams of base soap was weighed accurately to a well-washed beaker. A 100 ml of distilled water was added. It was boiled and then it was cold. The soap solution was transferred to a cleaned 500 ml separating funnel. 25 ml of 4N $H_2SO_4$was added. 100 ml of diethyl ether was added and it was stoppered. Then it was shacked by releasing the pressure from the valve. It was kept 20 minutes for settle. The water phase was drained out to another separating funnel. Another water wash was given with 50 ml of ether. The two ether phases were collected to the 1st separator. The water phase was drained out and it was left to settle. A dried and cooled round bottom flask was weighed. The ether extract was transferred carefully to the weighed flask, washing the separating funnel several times with fresh water. It was evaporated. The ether phase was recovered on a hot water bath, using sox let condensation.

About 3 ml of acetone was added. It was left again on a water bath. It was dried in an oven for 3 hours. Then, it was cold and weighed.

3.4 Determination of Total Anionic Detergents.

Apparatus

Electric balance, Volumetric flask, Graduated flask, Pipette, Titration vessel, Burette

Chemicals

Hyamine solutions (0.004M), Mixed indicator stock solution, Acid indicator solution, Chloroform, Phenolphthalein

Check sample: Rin powder

Procedure

Ten grams of the representative sample was weighed to the nearest 0.01 g. It was dissolved in about 150 ml of hot water. The solution was transferred quantitatively to a 1 litter graduated flask. Then it was diluted to volume with water.

Five mlliliters of this solution was pipetted out in to a titration vessel and it was diluted the volume to about 30 ml with water. A few drops of phenolphthalein indicator were added. (If required, neutralize with 0.1N sodium hydroxide to a pain pink color) 10 ml of acid indicator solution and 15 ml of chloroform were added. The stoppered vessel was shaked thoroughly. Then, it was titrated with Hyamine solution.

CHAPTER 04 RESULTS AND CALCULATIONS

4:1 Definitions

Stranded deviation (s)

Refers to a single analysis of the check sample under conditions that the analytical method is in control.

Target value

Is the long-term average of the results obtained from the check sample.

Warning limits

Are located at +2s and –2s above and below the target value.

Control limits

Are located at +3s and –3s above and below the target value.

Ucl – upper control limit

Uwl – upper warning limit

Lcl - lower control limit

Lwl – lower warning limit

4:2 Determination of Glycerol in crude Glycerin

4:2:1 Calculation of crude Glycerine

W = weight if the sample
N = Normality of NaOH
V_1 = volume of N/10 NaOH used (sample)
V_2 = volume of N/10 NaOH used (blank)

$$\% \text{ Glycerol} = \frac{(V1 - V_2) * N * 92.1 * 100}{1000 * W}$$

4:2:2 Check sample readings

Table 4. 1 20 observations of the training set for Glycerol

Sample No:	Strength of Glycerine
1	75.4
2	75.1
3	75.9
4	76.0
5	76.1
6	76.5
7	75.2
8	76.2
9	76.0
10	75.3
11	76.1
12	75.1
13	75.5
14	75.8
15	75.3
16	75.7
17	76.2
18	75.1
19	76.2
20	75.4

Average = 75.7

Standard deviation = 0.47

2s = 0.94

3s = 1.41

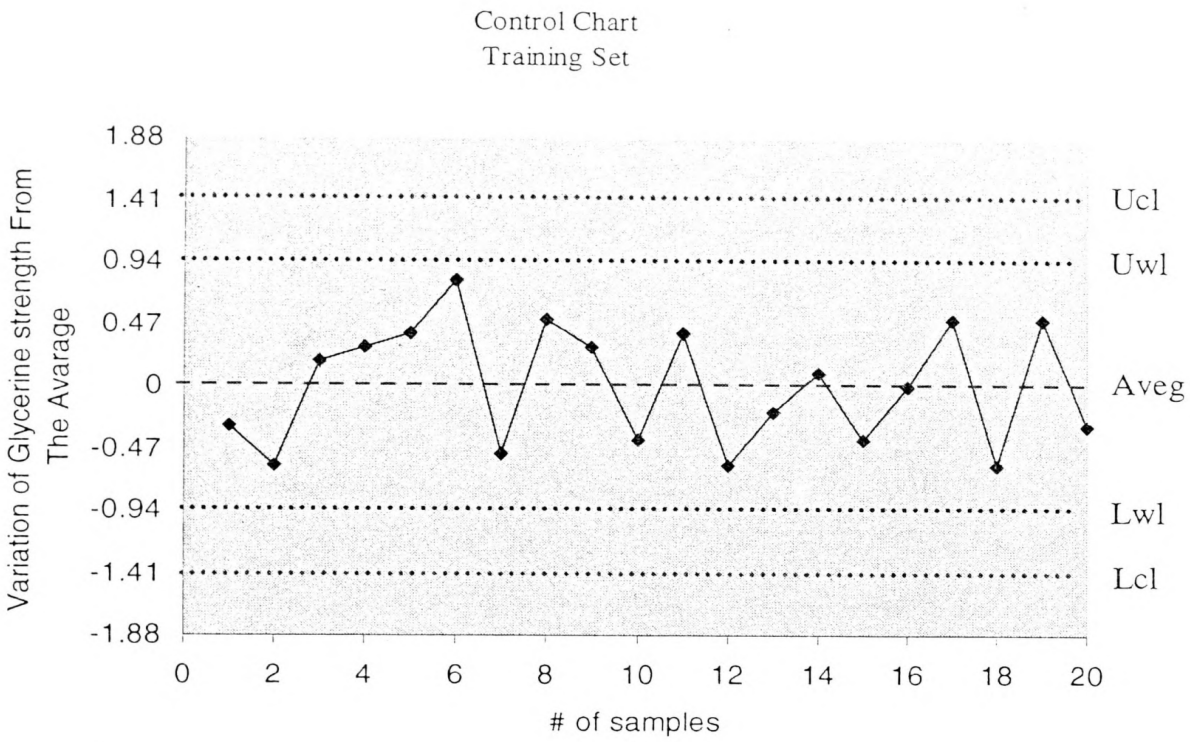


Figure 4.1 Control chart for training set for Glycerol

Table 4.2 Readings for continuous data (from 14/03/2005 to 10/06/2005)

Date	% Glycerol	Average
14/03/05	75.5	
15/03/05	75.6	
16/03/05	75.8	75.5
17/03/05	75.4	
18/03/05	75.2	
28/03/05	75.3	
29/03/05	75.9	
30/03/05	75.7	75.7
31/03/05	76.0	
01/04/05	75.4	
04/04/05	75.5	
05/04/05	76.1	
06/04/05	76.2	75.8
07/04/05	76.0	
08/04/05	75.2	
18/04/05	75.8	
19/04/05	75.6	
20/04/05	75.4	75.7
21/04/05	75.8	
22/04/05	75.7	
03/05/05	75.3	
04/05/05	75.9	75.8
05/05/05	76.0	
06/05/05	76.1	
09/05/05	75.2	
10/05/05	75.4	
11/05/05	75.6	75.4
12/05/05	75.7	
13/05/05	75.3	
16/05/05	75.8	
17/05/05	75.9	
18/05/05	76.1	75.9
19/05/05	76.0	
20/05/05	75.8	
30/05/05	75.6	
31/05/05	75.5	
01/06/05	75.4	75.5
02/06/05	75.6	
03/06/05	75.3	
06/06/05	76.1	
07/06/05	75.8	
08/06/05	75.4	75.7
09/06/05	75.3	
10/06/05	75.8	

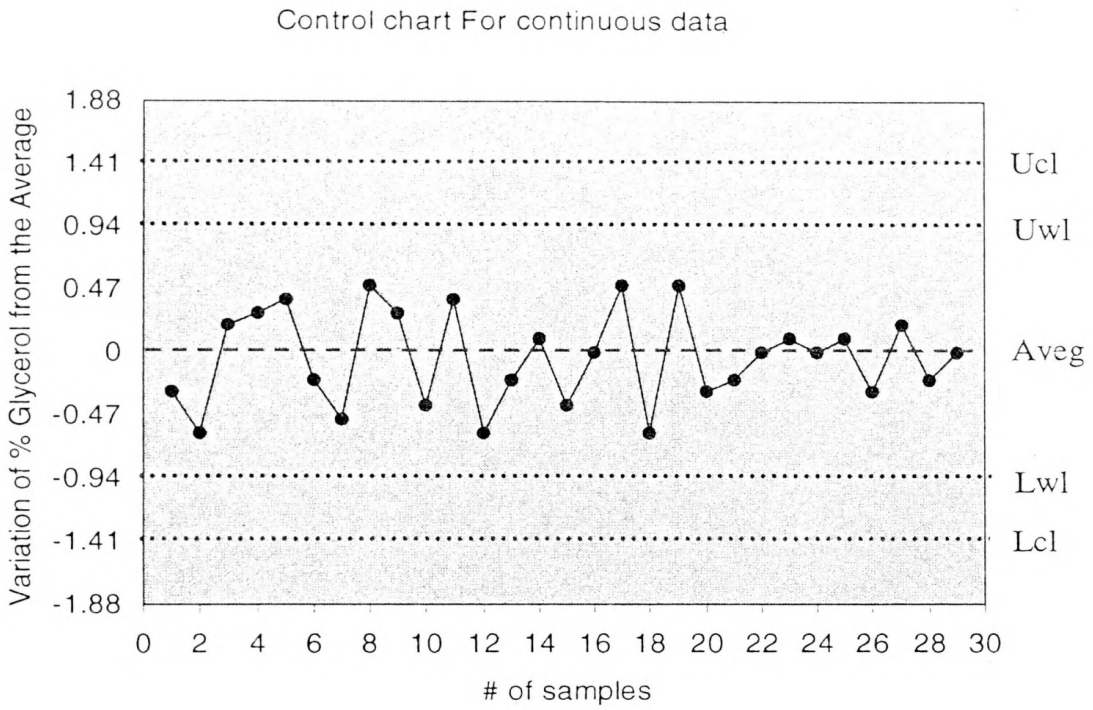


Figure 4.2 Control chart for continuous data for Glycerol

4:3 Determination of NaCl in soaps

4:3:1 Calculation of NaCl

N = Normality of $AgNO_3$
V = volume of $AgNO_3$
W = weight of sample

$$\% \text{ NaCl} = \frac{5.85 * N * V}{W}$$

$$\% \text{ KCl} = \frac{7.45 * N * V}{W}$$

4:3:2 Check sample readings

Table 4:3 20 observations of the training set for KCl

Sample no	% of Potassium Chloride (KCl)
1	99.7
2	99.8
3	99.7
4	99.9
5	99.6
6	99.8
7	99.5
8	99.7
9	99.9
10	99.8
11	99.8
12	99.7
13	99.9
14	99.6
15	99.8
16	99.7
17	99.9
18	99.6
19	99.7
20	99.5

Average = 99.7

Standard deviation = 0.13

2s = 0.26

3s = 0.39

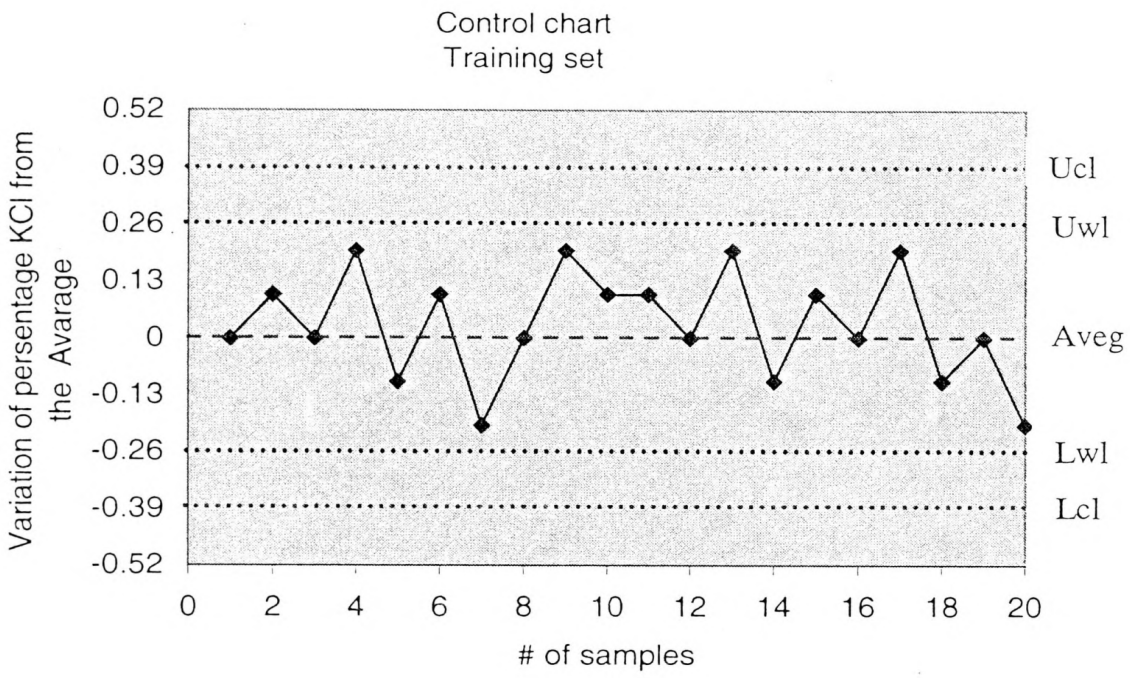


Figure 4.3 Control chart for training set for KCl

Table 4.4 Readings for continuous data (from 28/03/2005 to 03/06/2005)

Date	% KCl	Average
28/03/05	99.6	
29/03/05	99.5	
30/03/05	99.7	99.6
31/03/05	99.8	
01/04/05	99.6	
04/04/05	99.6	
05/04/05	99.8	
06/04/05	99.7	99.7
07/04/05	99.5	
08/04/05	99.9	
18/04/05	99.7	
19/04/05	99.5	
20/04/05	99.8	99.7
21/04/05	99.7	
22/04/05	99.6	
03/05/05	99.8	
04/05/05	99.8	
05/05/05	99.5	99.7
06/05/05	99.6	
09/05/05	99.7	
10/05/05	99.5	
11/05/05	99.5	99.6
12/05/05	99.8	
13/05/05	99.7	
16/05/05	99.9	
17/05/05	99.8	
18/05/05	99.7	99.7
19/05/05	99.5	
20/05/05	99.7	
30/05/05	99.6	
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01/06/05	99.8	99.7
02/06/05	99.5	
03/06/05	99.9	

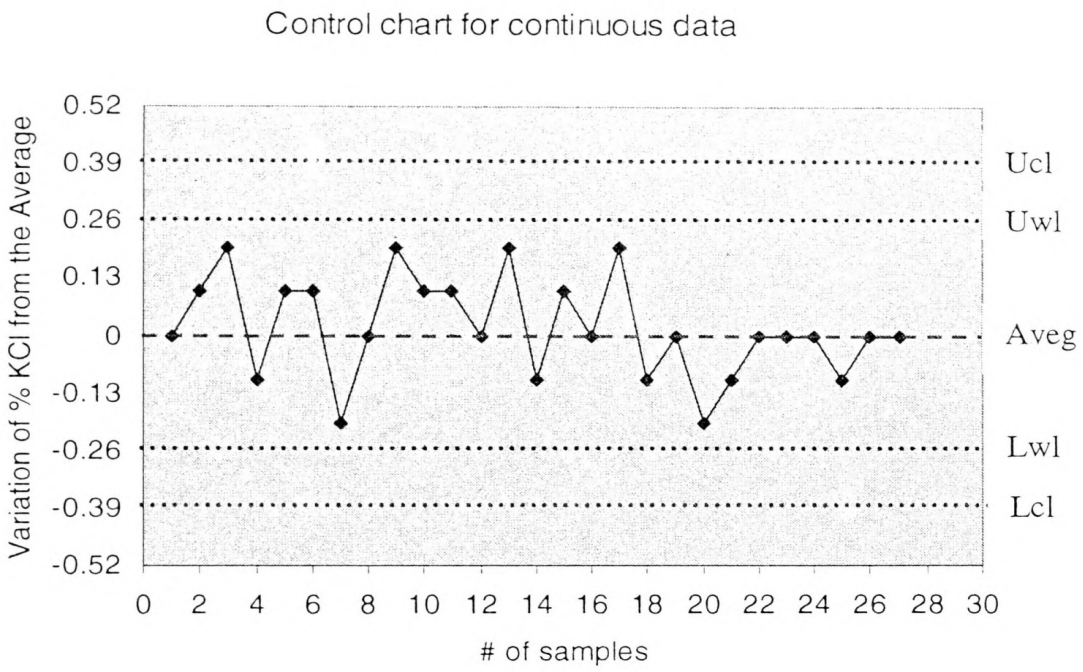

Figure 4.4 Control chart for continuous data for KCl

4.4 Determination of Total fatty matter (TFM)

4.4.1 Calculation of TFM

W = weight of the soap
W_1 = empty round bottom flask
W_2 = final weight of the flask with sample

$$\% \text{ TFM} = \frac{(W_2 - W_1) * 100}{W}$$

4.4.2 Check sample readings

Table 4.5 20 observations of the training set for KCl

# of samples	% TFM
1	61.1
2	61.3
3	61.2
4	61.3
5	61.0
6	60.9
7	61.4
8	61.2
9	61.1
10	61
11	60.8
12	60.9
13	61.1
14	61.2
15	61.4
16	61.3
17	60.8
18	60.9
19	61.3
20	61.2

Average = 61.1

Standard deviation = 0.2

2s = 0.4

3s = 0.6

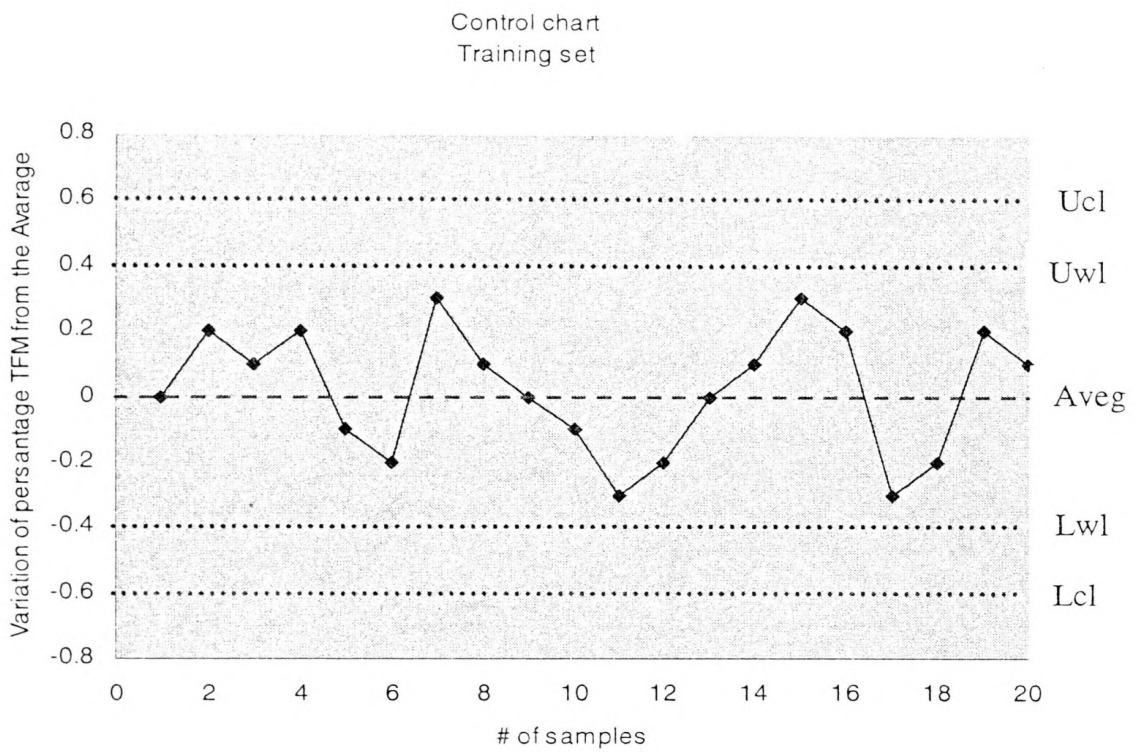

Figure 4.6 Control chart for training set for TFM

Table 4.6 Readings for continuous data (from 04/03/2005 to 03/06/2005)

Date	% TFM	Average
04/04/05	61.2	
05/04/05	61.2	
06/04/05	61.0	61.0
07/04/05	60.8	
08/04/05	60.9	
18/04/05	61.1	
19/04/05	61.2	
20/04/05	61.3	61.1
21/04/05	61.0	
22/04/05	60.8	
03/05/05	60.9	
04/05/05	61.2	61.1
05/05/05	61.1	
06/05/05	61.3	
09/05/05	60.9	
10/05/05	60.8	
11/05/05	61.1	61.1
12/05/05	61.2	
13/05/05	61.3	
16/05/05	61.1	
17/05/05	60.0	
18/05/05	61.2	60.9
19/05/05	61.2	
20/05/05	60.9	
30/05/05	61.1	
31/05/05	61.0	
01/06/05	61.2	61.1
02/06/05	60.9	
03/06/05	61.3	

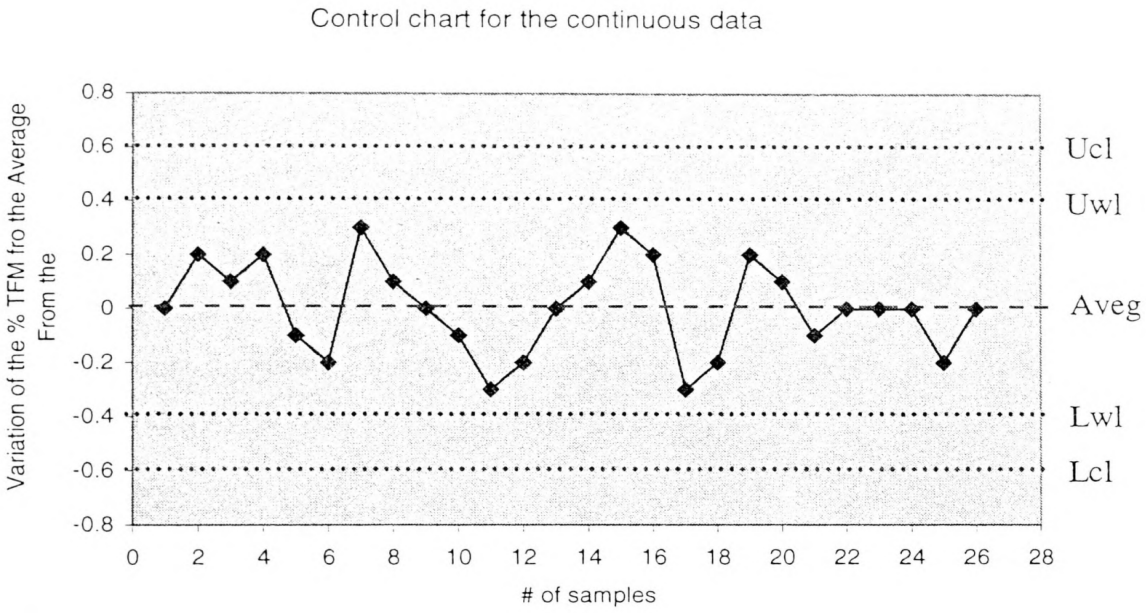

Figure 4.7 Control chart for continuous data for TFM

4.5 Determination of Total Anionic Detergent

4.5.1 Calculation of AD

$$AD = \frac{(V_2 - V_3) * M * MW * 100}{W * V_1}$$

W – Weight of sample

M – Molarity of Hygiene

MW – Molicular weight of cationic active matter

V_1 – Volume sample solution taken for titration

V_2 – Volume Hymine used for titration of SLS solution

4.5.2 Check sample readings

Table 4.7 20 observations of the training set for AD

# Of samples	% AD
1	14.1
2	14.3
3	14.2
4	14.4
5	14.5
6	14.1
7	14.2
8	14.6
9	14.0
10	14.4
11	14.6
12	14.5
13	14.2
14	14.1
15	14.6
16	14.3
17	14.5
18	14.2
19	14.5
20	14.6

Average = 14.3

Standard deviation = 0.19

2s = 0.38

3s = 0.57

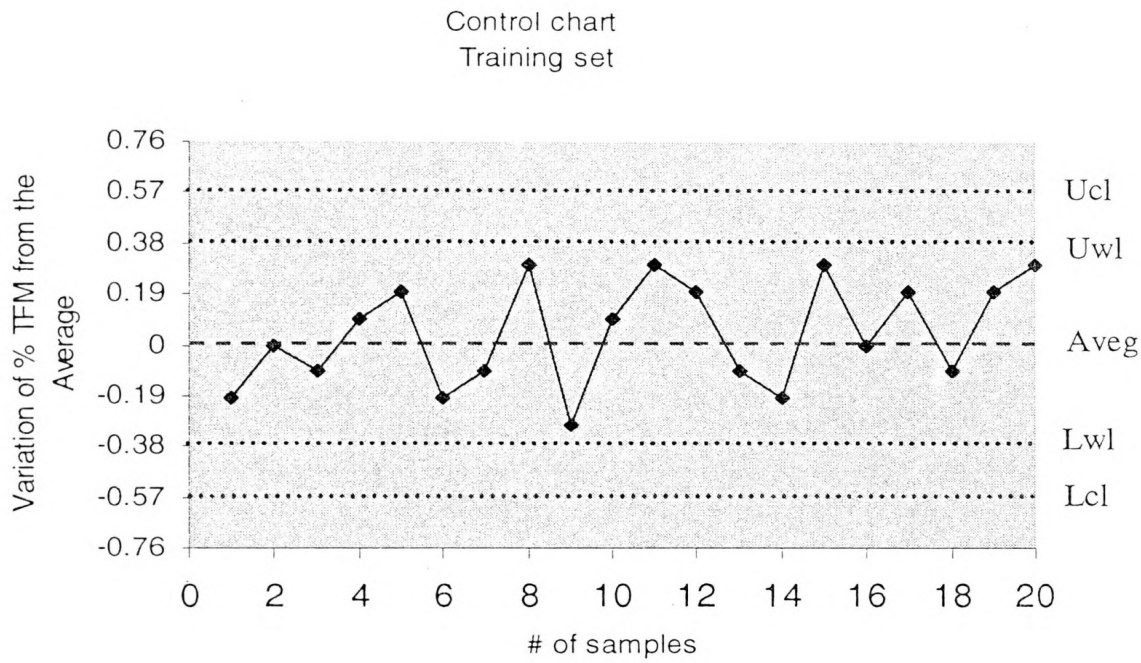

Figure 4.7 Control chart for training set for

Table 4.8 Readings for continuous data (from 04/03/2005 to 03/06/2005)

Date	% AD	Average
04/04/05	14.2	
05/04/05	14.4	
06/04/05	14.5	14.3
07/04/05	14.3	
08/04/05	14.3	
18/04/05	14.6	
19/04/05	14.3	
20/04/05	14.4	14.4
21/04/05	14.1	
22/04/05	14.6	
03/05/05	14.2	
04/05/05	14.5	14.3
05/05/05	14.4	
06/05/05	14.4	
09/05/05	14.2	
10/05/05	14.3	
11/05/05	14.6	14.4
12/05/05	14.5	
13/05/05	14.4	
16/05/05	14.3	
17/05/05	14.1	
18/05/05	14.5	14.3
19/05/05	14.1	
20/05/05	14.3	
30/05/05	14.3	
31/05/05	14.5	
01/06/05	14.1	14.3
02/06/05	14.2	
03/06/05	14.6	

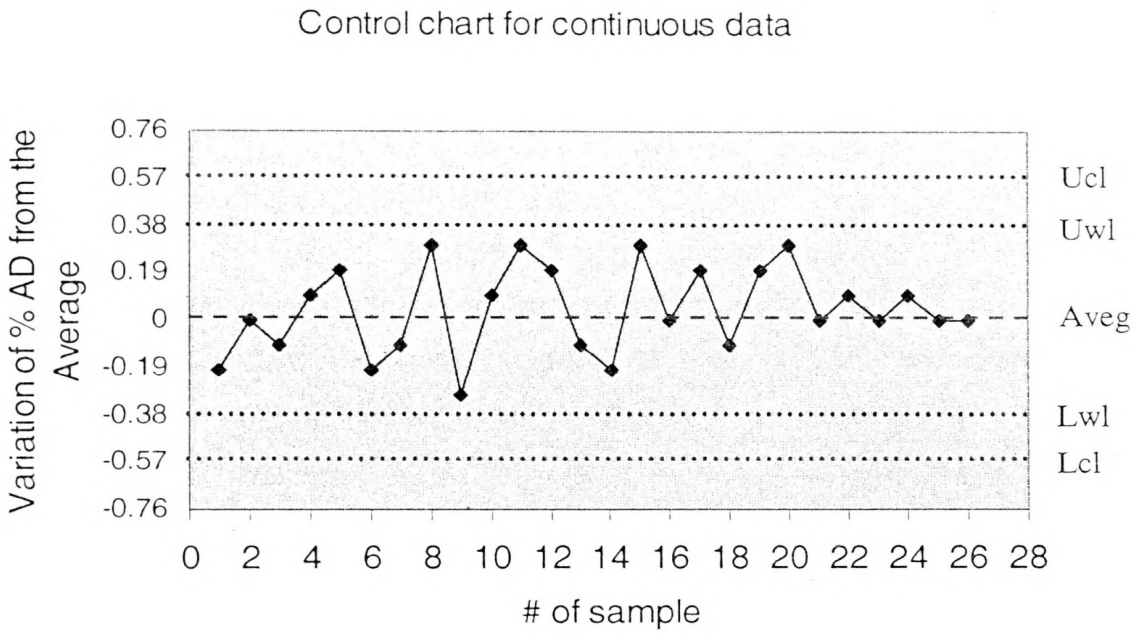

Figure 4.8 Control chart for continuous data for AD

CHAPTER 05 DISCUSSION AND CONCLUTION

5:1 Discussion

5:1:1 Use of the control chart

Once the training set was completed the control chart was ready to accept new observations from subsequent determinations of the check sample. New data entered in the control chart reveal whether or not the analytical determination is in control. For this decision we use the warning limits (2s) and the control limits (3s). From their definition it can statistically be expected that one out of every twenty observations will exceed the warning limits and one out of every thousand the control limits. As long as that is case we consider method in control. If however, the observations for the check sample exceed the warning or the control limits more frequently, then we conclude that the method is no longer in control. Generally speaking there are 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 is relevant , because the nature of the disturbance helps us to identify possible cases and take corrective action . When the case of the disturbance has been identified and repaired, a new determination of the check sample should fall within the warning limits and the analysis can be resume.

5:1:2 Consequence of out of control situation

5:1:2:1 Evaluation of the analytical method

If an out of control situation has occurred further analysis must be post pone until the case has been identified and removed. The check sample has deteriorated and no longer confirms to the original composition. If there is a failure in the instrumentation, we should test the equipment, readjust or replace malfunctioning parts, and verify proper operation. Thereafter run a check sample to assure that it's result falls within the warning limits.

5:1:2:2 Reporting analytical results preceding the out of control situation

When an out of situation is observed the regular analytical determinations of ordinary samples must be discontinues until the case of the out of control has been removed. The question remains, however, what must be done with the results of the analyses that have been performed since the previous determination of the check sample. If the tolerance for the analytical result is much wider than the variability of the method, i.e. the precision is more than adequate, than the analytical result can still be released. If the tolerance for the analytical results 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 reanalyzed.

5:1:3 Updating the control chart

5:1:3:1 New check sample

The control chart has been constructed on the basis of twenty observations from the training set, but the two parameters, the average and the standard deviation , can not be expected to remained the same infinitum. One clear case arises when a new check sample is introduced. Although the standard deviation of the analytical method is expect to remain the same , it is equally likely that the target value has changed. Analyze the new check sample at least six times over several days to determine the new target value.

5:1:3:2 Periodic update of the control chart after 25 new data points

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

If the remaining fresh results are evenly distributed around the target value within the existing control limits with no more than one point outside the control limits without and identified cause, then we add the data to the training set and the recalculate the average and the standard deviation from the total area if the fresh results are again evenly distributed around the target value , but two or more data exceed the control limit without clearly identified cause, then first calculate the average and the standard deviation from the total data. If the fresh data are not randomly distributed, but demonstrate a one sided bias or 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 can not be amalgamated with the data from the training set and the analytical method needs to be further investigated.

5:2 CONCLUTION

In the training period, for selected analytical determinations were used to check the in control or out of control . All the analytical determinations were in control

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Uniliver Method of analysis, volume B part 5 B. iv.b.11.1

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