



TOGETHER
for a sustainable future

OCCASION

This publication has been made available to the public on the occasion of the 50th anniversary of the United Nations Industrial Development Organisation.



TOGETHER
for a sustainable future

DISCLAIMER

This document has been produced without formal United Nations editing. The designations employed and the presentation of the material in this document do not imply the expression of any opinion whatsoever on the part of the Secretariat of the United Nations Industrial Development Organization (UNIDO) concerning the legal status of any country, territory, city or area or of its authorities, or concerning the delimitation of its frontiers or boundaries, or its economic system or degree of development. Designations such as “developed”, “industrialized” and “developing” are intended for statistical convenience and do not necessarily express a judgment about the stage reached by a particular country or area in the development process. Mention of firm names or commercial products does not constitute an endorsement by UNIDO.

FAIR USE POLICY

Any part of this publication may be quoted and referenced for educational and research purposes without additional permission from UNIDO. However, those who make use of quoting and referencing this publication are requested to follow the Fair Use Policy of giving due credit to UNIDO.

CONTACT

Please contact publications@unido.org for further information concerning UNIDO publications.

For more information about UNIDO, please visit us at www.unido.org



UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

UNIDO PROJECT NUMBER: MP/NIR/07/002

CONTRACT NUMBER: 16002232

REVISED REPORT (3 OF 7)

**DETAILED CHEMICAL ANALYSIS OF ACTIVATOR
(SOFTENER CONCENTRATE)**

SUBMITTED BY

**BEST ENVIRONMENTAL SOLUTION TOOLS LIMITED
2 TEWOGBADE STREET, OJODU, P. O. BOX 4439, IKEJA
LAGOS. NIGERIA**

NOVEMBER 2011

ACKNOWLEDGEMENTS

The Management and Staff of Best Environmental Solution Tools Limited hereby express our profound gratitude to the United Nations Industrial Development Organization (UNIDO), Vienna International Centre, Austria for giving us the opportunity to undertake the detailed chemical analysis of Activator (Softener Concentrate).

Our appreciation also goes to the Nigeria National Ozone Officer, Mr. Kasimu Bayero and the National Solvent Officer, Mr. Charles Ikeah for their cooperation towards successful execution of this project.

The positive and constructive contribution of the UNIDO Project Management Team in enriching the quality of the work towards the production of this revised version of the report is gratefully appreciated by the Laboratory Analysis Team of Best Environmental Solution Tools Limited.

TABLE OF CONTENTS

Title	Page
Title Page	1
Acknowledgement	2
Table of Contents	3
List of Tables	4
Abbreviations and Acronyms	6
EXECUTIVE SUMMARY	7
CHAPTER ONE: INTRODUCTION	8
1.1 Background	8
1.2 Aims and Objectives of the Study	9
1.3 Scope of Analytical Work and Services	9
1.4 Analysis of Activator	10
CHAPTER TWO: APPLICABLE PARAMETERS REQUIREMENT	11
2.1 Quality Parameters	11
CHAPTER THREE: MATERIALS, METHODS & ANALYTICAL PROCEDURES	13
3.1 Sample Collection	13
3.2 Sample Preservation and Storage	13
3.3 Analytical Procedures	13
3.3.1 pH	13
3.3.2 Colour	13
3.3.3 Specific Gravity	14
3.3.4 Phase	14
3.3.5 Odour	14
3.3.6 Solubility Rating	15
3.3.7 Reactivity Rating	15

3.3.8	Evaporation Rating	15
3.3.9	Flammability	16
3.3.10	Boiling Point	16
3.3.11	Freezing Point	17
3.3.12	Metals	17
3.3.13	Total Sulphur	18
3.3.14	Oil and Grease	18
3.3.15	Total Hydrocarbons	19
3.3.16	Polyaromatic Hydrocarbons	19
3.3.17	Polychlorinated Biphenyls	20
3.3.18	BTEX	20
3.4	Quality Assurance Procedure	21
CHAPTER FOUR: RESULTS AND DISCUSSION		22
4.1	Physico-Chemical Properties	22
4.2	Heavy Metals	23
4.3	Organic Compounds Analysis	23
4.3.1	Oil & Grease and Total Hydrocarbons	23
4.3.2	Polyaromatic Hydrocarbons	24
4.3.3	Polychlorinated Biphenyls	25
4.3.4	BTEX	27
CHAPTER FIVE: CONCLUSION AND RECOMMENDATION		28
5.1	Conclusion	28
5.2	Recommendation	29
APPENDICES		30

LIST OF TABLES

Table	Title	Page
Table 2.1	Parameters Requirement for Activator	11
Table 4.1	Physico-Chemical Properties of Activator	22
Table 4.2	Heavy Metal Levels in Activator	23
Table 4.3	Oil & Grease and THC Levels of Activator	24
Table 4.4	Correlation Coefficients for Selected PAH Standards	24
Table 4.5	Polyaromatic Hydrocarbons in Activator	25
Table 4.6	Correlation Coefficients for Selected PCB Standards	26
Table 4.7	Polychlorinated Biphenyls in Activator	26
Table 4.8	Correlation Coefficients for Selected BTEX Standards	27
Table 4.9	BTEX in Activator	27

ABBREVIATIONS AND ACRONYMS

AAS	Atomic Absorption Spectrophotometer
ASTM	American Society for Testing and Materials
BEST	Best Environmental Solution Tools Limited
BTEX	Benzene, Toluene, Ethyl Benzene and Xylene
CFC	Chlorofluorocarbon
CTC	Carbon Tetrachloride
ECD	Electron Capture Detector
FID	Flame Ionization Detector
HCFC	Hydrochlorofluorocarbon
GC	Gas Chromatograph
MIBK	Methyl Isobutyl Ketone
NOO	National Ozone Office
ODS	Ozone Depleting Substance
PAH	Polyaromatic Hydrocarbon
PCB	Polychlorinated Biphenyl
QA	Quality Assurance
QC	Quality Control
TCA	Trichloromethane
THC	Total Hydrocarbons
UNEP	United Nations Environment Programme
UNIDO	United Nations Industrial Development Organization
UV	Ultra Violet
VIS	Visible

EXECUTIVE SUMMARY

Sample of Activator (Softener Concentrate) was collected from Mr. Charles Ikeah at the Federal Ministry of Environment, Abuja, Nigeria in January 2011 and analyzed for priority physical properties, chemical elements and organic parameters listed in the scope of analysis of the contract document.

Activator was preserved in accordance with international practice by keeping it at less than 4°C in the laboratory until ready for analysis. ASTM methods and modified procedures contained in BEST laboratory test methods were adopted in the analysis of Activator. Good laboratory practice encompassing standard operating procedures for quality assurance and quality control were strictly adhered to in the analysis of the sample.

The study revealed that Activator was alkaline with pH value of 11.70, nonflammable with specific gravity of 1.001, colourless, miscible with water, non-reactive and stable in nature under normal conditions of temperature and pressure. Its boiling point was about 100°C while its freezing point was almost 0°C. The levels of toxic elements and organic pollutants in Activator, namely: heavy metals, oil and grease, total hydrocarbons, polynuclear aromatic hydrocarbons, polychlorinated biphenyls and BTEX were either not detected or extremely low in concentrations.

Essentially, any formulated cleaning product should, therefore, not be strongly acidic ($\text{pH} < 2$), not strongly alkaline ($\text{pH} > 12$), not flammable, not lighter than water, not volatile and not reactive. In addition, toxic elements and organic pollutants must be below permissible levels for human use as well as the cleaning equipment and should not have adverse effects on the environment.

In order to further confirm our findings on the concentrations of quality parameters described in this report for Activator, another study may be undertaken to determine its impact on the environment by carrying out aquatic toxicity tests on the sample.

CHAPTER ONE

INTRODUCTION

1.1 Background

In May 1981, the United Nations Environment Programme (UNEP) Governing Council launched negotiations on an international agreement to protect the ozone layer and, in March 1985 after years of difficult negotiations, the 1985 Vienna Convention for the Protection of the Ozone Layer was adopted. In September 1987, efforts to negotiate binding obligations to reduce the use of ODS led to the adoption of the “Montreal Protocol on Substances that Deplete the Ozone Layer” which was signed as a binding instrument on the parties to phase-out the use of ozone depleting substances (ODS) in their respective countries. The Vienna Convention on the Protection of the Ozone Layer and the Montreal Protocol on Substances that Deplete the Ozone Layer are Multilateral Environmental Agreements designed to protect the ozone layer through the phase-out of ozone depleting substances which include CFCs, HCFCs, CTC, TCA, halons, methyl bromide. These substances are grouped under Annexes I, II, III and IV of the Montreal Protocol. Solvent sector is one of the various ODS sectors in the implementation of the Montreal Protocol.

Solvents are liquids with the ability to dissolve, suspend or extract other materials without chemically changing those materials or themselves. They have been used in many industrial areas over many years, to remove grease from metals and printed circuit boards, produce intermediates for pharmaceuticals and agrochemicals, chlorinated polymers and polyurethane foam materials and to remove paints and coatings or in dry cleaning. Solvents are freely soluble and can easily be reconcentrated and recycled. Some, like methyl chloroform (TCA) and carbon tetrachloride (CTC) are considered to be the substances chiefly responsible for stratospheric ozone depletion. They contribute to the man-made greenhouse effect and because they are also possible health hazards for people working with them as well as being harmful for the environment, their substitution with alternative products has become imperative.

UNIDO has been very active in the solvent sector right from the beginning of its work with the Multilateral Fund for the Montreal Protocol of the Vienna Convention on substances that deplete

the ozone layer. The organization has prepared solvent and process agent projects in several countries including Nigeria. This project is part of UNIDO's intervention in the CTC phase-out programme in the fourth tranche of the Terminal ODS Phase-Out Umbrella Project in the Solvent Sector in Nigeria. CTC while being a good, low-cost solvent is classified as toxic and carcinogenic. CTC can be avoided or substituted by other non-ODS process solvents. The parts cleaners in Nigeria are small scale enterprises and it has been found out that it would be difficult and very expensive for them to continue to import these alternative cleaning solvents for their use and will therefore adversely affect their operations. Some of them have the capability to compound these cleaning solvents given some guidance on the analytical parameters of the chemicals. The seven solvent cleaning solutions to be analyzed, namely: Energizer (Rinse Agent Concentrate), Ultrasonic Cleaning Solution Concentrate, Activator (Softener Concentrate), Carbon Burster (Carbon Cleaning Solution), Super Energizer (Degreaser Concentrate), Parts Cleaner and Miracle Cleaner, are some of the alternatives to CTC.

1.2 Aims and Objectives of the Study

The aim of the project is to finalize CTC phase-out in Nigeria. The main objective of the analysis is to enable formulators in compounding the solvent solutions that will be readily available in Nigeria in the optimization of the use of the ultrasonic technologies as an alternative to ozone depleting solvents thus creating markets for local solvent formulators and making the parts cleaning sector more sustainable. Another objective of the analysis is to ensure that the solvents are environment friendly, non-toxic and that they do not pose health risks, especially to the personnel that would use them in their operations.

1.3 Scope of Analytical Work and Services

The services to be delivered in order to achieve the aims and objectives of this study include:

- Carrying out a detailed chemical analysis of the seven solvent cleaning solutions;
- Presenting data analysis of the results to ensure correctness and acceptability with National and International Standards for solvent formulation quality control checks;
- Submitting data for cross-check by the National Ozone Office Unit; and
- Preparing a comprehensive report on the methodology employed and the results obtained.

The Scope of Analysis includes, but not limited to, the following parameters in establishing quality control checks for solvent cleaning solutions:

- Carrying out elemental analysis for the determination of the levels of magnesium and sulphur in the solvent cleaning solutions;
- Determining the levels of the following heavy metals: cadmium, iron, lead, chromium, zinc, copper, nickel, mercury, arsenic, cobalt, vanadium and manganese;
- Conducting necessary laboratory analyses to determine the oil and grease, total hydrocarbon, polyaromatic hydrocarbon, BTEX, and PCB content of the cleaning solutions;
- Providing the following physical properties of the solvent solutions through appropriate laboratory tests: pH, colour, odour, phase, specific gravity, solubility rating, reactivity rating, evaporation rating, flammability, boiling point, freezing point; and
- Collaborating with the National Ozone Office (NOO) in cross-checking the data obtained from the analyses.

1.4 Analysis of Activator

Activator belongs to the family of cleaner solutions in an alkaline medium that has general application in homes and industrial environment. UNIDO commissioned Best Environmental Solution Tools Limited to carry out detailed chemical analysis of Activator. The results obtained from the laboratory analysis of the chemical are presented in this report and will allow solvent formulators to be informed of the levels of the parameters permissible in their chemical formulations.

CHAPTER TWO

APPLICABLE PARAMETERS REQUIREMENT

2.1 Quality Parameters

The scope of study listing required parameters for physical and chemical analysis of Activator (Softener Concentrate) are presented in Table 2.1.

Table 2.1: Parameters Requirement for Activator (Softener Concentrate)

S/N	Required Parameters
1	pH
2	Colour
3	Specific Gravity
4	Phase
5	Odour
6	Solubility Rating
7	Reactivity Rating
8	Evaporation Rating
9	Flammability
10	Boiling Point
11	Freezing Point
12	Cadmium (Cd)
13	Iron (Fe)
14	Lead (Pb)
15	Chromium (Cr)
16	Magnesium (Mg)
17	Zinc (Zn)
18	Copper (Cu)
19	Nickel (Ni)
20	Mercury (Hg)
21	Arsenic (As)

S/N	Required Parameters
22	Cobalt (Co)
23	Vanadium (V)
24	Manganese (Mn)
25	Total Sulphur (S)
26	Oil and Grease (O & G)
27	Total Hydrocarbons (THC)
28	Polyaromatic Hydrocarbons (PAH)
29	Benzene/Toluene/Ethylbenzene/Xylene (BTEX)
30	Polychlorinated Biphenyls (PCB)

CHAPTER THREE

MATERIALS, METHODS AND ANALYTICAL PROCEDURES

3.1 Sample Collection

Representative sample of Activator (Softener Concentrate) was collected from the big container into a 4-litre container and transported to the laboratory in a cooling medium of ice chest.

3.2 Sample Preservation and Storage

Activator was preserved in accordance with international practice by keeping it at less than 4°C in the laboratory until ready for analysis.

3.3 Analytical Procedures

Laboratory methods adopted in the analysis of Activator using ASTM and modified procedures in accordance with BEST laboratory test methods are presented below.

3.3.1 pH

An electrometric method was used with the aid of an ATI-Orion Model 290A pH meter to determine the pH of the Activator sample. The pH meter was standardized with three buffer solutions of pH units 4.01, 7.00 and 10.01 before measuring that of Activator. The instrument was also checked by measuring pH of distilled water. The electrode was washed and dried. The sample cup was also washed three times with distilled water and allowed to dry. Activator was allowed to thaw to room temperature before taking the pH measurement. The cup was filled with a portion of the sample dipping the electrode inside it to obtain the pH value of Activator. The meter was calibrated again with the three buffer solutions.

3.3.2 Colour

Lovibond Colour Comparator was used to determine the colour of Activator sample based on visual comparison with permanently coloured glass standards. The three cylindrical glass sample jars of the comparator were half-filled with Activator at the centre and the outer ones filled with distilled water. Activator was viewed through a prism that brings it and the colour standards into adjoining fields of view. The two discs containing the colour standards were rotated by turning

the control knobs on the front of the comparator until the colour of the sample fell between two standards which are 0.5 apart, or until it exactly matched one of the colour standards. The reading, given directly as ASTM colour, was then taken from the scale on the control knobs and the colour recorded for Activator.

3.3.3 Specific Gravity

Rapid determination of the specific gravity of Activator sample was achieved by the use of a hydrometer. Activator was gently poured into a dry hydrometer cylinder without splashing so as to avoid formation of air bubbles. The cylinder was placed vertically in a water bath, carefully stirring the contents of the cylinder until the temperature was getting close to 25°C at which the hydrometer was slowly and carefully lowered into the sample and then released. After the hydrometer was still and floated freely away from the walls of the cylinder, the gravity was read at the point at which the surface of the sample apparently cut the hydrometer scale as the distorted ellipse became a straight line. The specific gravity, at 25/25 C, of Activator was then calculated as the observed gravity value + correction factor 0.021.

3.3.4 Phase

This was determined by visual examination of the Activator sample in a 100-ml beaker at room temperature and recorded as either solid, liquid or gaseous, which are the three phases of matter in the scientific world.

3.3.5 Odour

The method employed for determining odour involves a procedure for observing the characteristic odour of Activator sample. This was carried out by dipping 25 by 75-mm strips of a qualitative filter paper to a depth of about 50mm into the sample and reference standard odour each contained in a 50-ml beaker. The odour of the two materials on the wet filter papers were compared until the odour of a standard that is similar to the odour of the sample was obtained and consequently recorded as the characteristic odour of Activator.

3.3.6 Solubility Rating

This is the measure of the ability of the Activator sample to dissolve in a solvent. It was determined by testing the solubility of the sample in water. At room temperature, about 10-ml of Activator was taken and transferred into a 250-ml conical flask and portions of distilled water were slowly added with increased mixing and agitation, observing whether the two phases were miscible with one another or not. The addition of the water solvent was continued until about 100-ml water was added to the Activator in the conical flask. Formation of a clear solution with no sign of cloudiness or precipitation indicates that the sample is miscible in water and on the other hand, if the resulting mixture is colloidal, the sample is therefore immiscible in water.

3.3.7 Reactivity Rating

Reactivity rating was determined by mixing the Activator sample with zinc oxide. 25g zinc oxide was mixed with 50-ml sample with the use of a mechanical mixer by alternate additions of the oxide and the liquid forming a fairly stiff paste at room temperature which was reduced to a viscous liquid by gradually working on the remainder of the sample. The consistency of the fresh liquid was determined by visual inspection with a spatula. The liquid was transferred into a friction-top can, filled to about 0.5 in. of the top with lid tightly inserted and stored at room temperature. After 24 hours of storage, the liquid in the can was thoroughly mixed bringing it to 25°C and the consistency determined. The sample was graded as nonreactive (stable), slightly reactive (slightly unstable), moderately reactive (moderately unstable) or reactive (very unstable) based on comparison of the consistencies of the fresh sample and aged sample.

3.3.8 Evaporation Rating

A thin-film evaporometer was used to determine the rate of evaporation of the Activator sample. The instrument was assembled and conditioned by placing the filter paper disk on the wire support threading the hook through a small hole in the centre of the paper. The hook was attached to the steel spring below the sighting disk with the paper and the paper support hanged therefrom. The evaporometer and cabinet doors were closed and allowed to equilibrate. The air flow rate was then adjusted to about 20 litres/min. At room temperature, the wire mesh bracket was raised until the bottom of the disk support rested lightly on it. 1ml of Activator was withdrawn into the hypodermic syringe and inserted into the small opening on the right hand side

of the equipment. The sample was applied drop by drop at 10-second intervals starting the timer as the first drop hit the disk. The wire mesh bracket was then lowered from the disk support. First reading was obtained after 40 seconds and then every 20 seconds recording the time and the scale reading. The timer was stopped when the filter paper returned to the original unloaded position. Percent evaporated was plotted against time drawing a smooth curve through the points. Relative evaporation rate was calculated from the 90% evaporated times for the sample, relative to that of butyl acetate with a value of 1.0.

3.3.9 Flammability

This is the tendency of a sample either to extinguish or to spread a flame after ignition and it refers to how easily a material will burn or ignite, causing fire or combustion. The degree of flammability of Activator sample was carried out through fire testing. Activator was introduced onto a bunsen flame in drops. The effect of the dripped particles of the sample was monitored and observed for flammability ratings. The degree of flammability was rated as 0 for non-flammable sample that does not burn but extinguish the fire, 1 for slightly flammable sample that must be preheated before it can ignite, 2 for moderately flammable sample that must be moderately heated or exposed to relatively high ambient temperatures before it can ignite, 3 for flammable sample that can ignite under almost all temperatures or 4 for highly flammable sample that will rapidly vapourize, readily dispersed in air and burn instantly.

3.3.10 Boiling Point

Boiling point apparatus was used to determine the boiling point of the Activator sample. 50-ml sample was transferred into the boiling flask and anti-bumping granules added. A calibrated thermometer was inserted through the side and a clean condenser was attached to the flask. The flask was mounted on wire gauze supported by a stand and held in place by a clamp. The cooling water inlet and outlet tubes were connected to the condenser. The condenser water and the electric heater were turned on. The contents of the flask was strongly heated initially and then slowly until effervescence was obtained, at which the temperature that was observed to be constant over a period of time being recorded as the boiling point of Activator at the prevailing barometric pressure.

3.3.11 Freezing Point

A freezing point tube was used to determine the freezing point of the Activator sample. The sample was cooled slowly with constant agitation in a freezing point tube until crystallization occurred. The freezing point apparatus was assembled by fitting the smaller test tube with a two-hole cork stopper with the thermometer inserted in the centre hole and the shaft of the stirrer through the other hole. Small layer of absorbent glass wool pad was placed in the bottom of the bigger test tube. The smaller test tube was inserted up to the lip into a cork stopper that fitted into the mouth of the bigger test tube. The cooling bath was filled with ice water. Sample was transferred into the smaller tube with the cork carrying the thermometer and the stirrer inserted. The sample in the smaller tube was cooled in ice bath with continuous and vigorous stirring until a constant temperature was reached and recorded as the freezing point of Activator.

3.3.12 Metals

The levels of the heavy metals were determined by spectrophotometric absorption of analyte metal ions through aspiration of Activator sample extract using Perkin Elmer AAS 3100 Atomic Absorption Spectrophotometer (AAS). Prior to aspiration of sample extract, calibration and standardization of the equipment was carried out by preparing six 100-ml aliquots of standard solutions of the metal of interest. The AAS was turned on and wavelength set to the value where maximum absorption occurs for the metal of interest and the instrument allowed to warm up for about 30 minutes and the slit width set. Air and acetylene pressures were adjusted and the burner was ignited. Distilled water was aspirated to rinse the atomizer chamber. Standard solutions were aspirated and necessary adjustments made to obtain maximum absorption; recording corresponding instrument readings from where a standard calibration curve is obtained with absorbance plotted against concentrations. For determination of the sample, 100-ml Activator was extracted using HNO₃ (1 + 1) solution. A hollow cathode lamp of the element of interest was also aligned in the Perkin Elmer atomic absorption spectrophotometer. The monochromator was adjusted to the appropriate wavelength and the required slit width for the metal of interest. With the correct fuel and oxidant setting for the flame, the burner was positioned for maximum absorption and stability. Absorbance of blank and standard samples was used to plot a calibration curve from where the concentration of the metal in the sample was determined. The levels of the following heavy metals were determined in the Activator sample: cadmium, iron, lead, chromium,

manganese, zinc, copper, nickel, mercury, arsenic, cobalt, vanadium and manganese.

3.3.13 Total Sulphur

This was determined by the use of Schöniger oxygen flask technique involving combustion of sulphur in the Activator sample to sulphuric acid which was determined by titration with barium chloride solution using sulphonazo indicator. 10-ml Activator was rapidly transferred into a laboratory capsule which contains a small piece of folded filter paper as absorbent. The cover of the capsule was quickly replaced and wrapped with more absorbent filter paper. The capsule was inserted in the platinum sample carrier. 50-ml of distilled water was transferred into the Schöniger flask and 1.0ml of hydrogen peroxide was added. The inside of the flask was flushed with a rapid flow of oxygen for 1 min. The stopper of the combustion flask was removed, the filter paper fuse ignited and the stopper containing the burning paper quickly inserted into the oxygen-filled combustion flask. The stoppered flask was inverted immediately to form a tight seal around the stopper. The combustion process was followed to completion with the inverted flask allowed to cool for few minutes. The flask was vigorously shaken to absorb the combustion products and allowed stand for about an hour. The inside of the flask was washed with distilled water and contents transferred into a conical flask and heated to boiling until about 10ml was left. This was allowed to cool to room temperature with sulphonazo indicator added and titrated against 0.01M barium chloride solution. The reading was taken at the end point when there was a distinct change from red to blue which was stable for about 5 minutes. The sulphur content was then determined by calculation using the titre value.

3.3.14 Oil and Grease

Quantity of oil and grease present in Activator sample was determined by partition gravimetric method involving extraction of a portion of the sample with an organic solvent followed by evaporation of the solvent in a tared flask and the residue in the flask weighed. 100ml sample was acidified to $\text{pH} < 2$ and serially extracted three times with n-hexane in a separatory funnel. The extract was dried over sodium sulphate. The solvent was distilled from the extract in a water bath, cooled in a desiccator for about one hour and weighed. The oil and grease in Activator was calculated from the amount of residue divided by the amount of sample.

3.3.15 Total Hydrocarbons

100-ml Activator sample was taken and spiked with acenaphthene internal standard. This was subsequently extracted with 100ml of Analar grade MIBK (methyl isobutyl ketone). The extract was allowed to settle, centrifuged and then decanted. The supernatant was injected for GC analysis. GC analysis for total hydrocarbons was carried out by a high resolution HRGC MEGA 2 Series (FISONS Instrument) Gas Chromatograph equipped with a flame ionization detector. A 30m length, 0.25 μ m film thickness and 0.25mm internal diameter capillary column was used and the peak areas were analyzed with an SRI Model 203 Peak Simple Chromatography Data System. The column temperature was 60°C for 2 min to 300°C programmed at 12°C/min. Nitrogen was used as carrier gas with a pressure of 37psi. Hydrogen and air flow rates were 9psi and 13psi, respectively. Injector port and detector temperatures were 250°C and 320°C, respectively. 2 μ l of sample extract was injected. In addition, known weights of Analar grade alkene standards spiked with known weight of acenaphthene internal standard were mixed and used for both qualitative identification and quantitative analyses.

3.3.16 Polyaromatic Hydrocarbons

100-ml Activator sample was taken and spiked with acenaphthene internal standard. This was subsequently extracted with 100ml of Analar grade MIBK (methyl isobutyl ketone). The extract was allowed to settle, centrifuged and decanted. This was further eluted with 200ml of 1:3 hexane/dichloromethane to elute the aromatic fraction. The eluate was concentrated over hydrogen and used for analysis by gas chromatography. GC analysis of the sample for polynuclear aromatic profile was carried out by a high resolution HRGC MEGA 2 Series (FISONS Instrument) Gas Chromatograph equipped with a flame ionization detector. A 30m length, 0.25 μ m film thickness and 0.25mm internal diameter capillary column was used and the peak areas were analyzed with an SRI Model 203 Peak Simple Chromatography Data System. The column temperature was 98°C for 1min to 300°C programmed at 8°C/min. Nitrogen was used as carrier gas with a flow rate of 37psi. Hydrogen and air flow rates were 12psi and 15psi, respectively. Injector port and detector temperatures were 250°C and 320°C, respectively. 2 μ l of sample was injected. Known weights of Analar grade standards of polynuclear aromatic hydrocarbons, namely; naphthalene, phenanthrene, benzo(b)fluoranthene and benzo(g,h,i)perylene, spiked with known weight of acenaphthene internal standard was mixed and used for both qualitative and quantitative analyses

of the aromatic contents. 2µl of sample was injected. The following polyaromatic hydrocarbons were determined in Activator: naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, benzo(a)anthracene, chrysene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, indeno(1,2,3-cd)pyrene, dibenzo(a,h)anthracene, and benzo(g,h,i)perylene.

3.3.17 Polychlorinated Biphenyls

100-ml Activator sample was taken and spiked with acenaphthene internal standard. This was subsequently extracted with 100ml of Analar grade MIBK (methyl isobutyl ketone). The extract was allowed to settle, centrifuged and then decanted. This was further eluted with 200ml of 1:3 hexane/dichloromethane to elute the aromatic fraction. The eluate was concentrated over hydrogen and used for analysis by gas chromatography. GC analysis of the sample for polychlorinated biphenyls was carried out by a high resolution Perkin Elmer Auto System Gas Chromatograph, fitted with a Ni-63 electron capture detector. A 60m length, 0.25µm film thickness and 0.25mm internal diameter capillary column was used and the peak areas were analyzed with an SRI Model 203 Peak Simple Chromatography Data System. The chromatographic conditions for analysis of sample extract are: initial oven temperature 100°C, initial temperature ramp 15°C/min to 150°C, second temperature ramp 3°C/min to 265°C, carrier gas helium at 1 ml/min and make-up gas nitrogen at 40 ml/min. 2µl of sample was injected. Known weights of Analar grade standards of selected polychlorinated biphenyl standards, namely: 2,4'-dichlorobiphenyl; 2,2',3,4,5'-pentachlorobiphenyl; 2,2',3,3',4,4',5,6-octachlorobiphenyl and 2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl, spiked with known weight of acenaphthene internal standard was mixed and used for both qualitative and quantitative analyses. 2µl of sample was injected. The following polychlorinated biphenyls were determined in Activator: 2,4'-dichlorobiphenyl; 2,2',5-trichlorobiphenyl; 2,3',3,5'-tetrachlorobiphenyl; 2,2',3,4,5'-pentachlorobiphenyl; 2,2',3,3',4,4'-hexachlorobiphenyl; 2,2',3,3',4,4',5-heptachlorobiphenyl; 2,2',3,3',4,4',5,6-octachlorobiphenyl; 2,2',3,3',4,4',5,5',6-nonachlorobiphenyl and 2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl.

3.3.18 Benzene/Toluene/Ethylbenzene/Xylene

100-ml Activator sample was taken and spiked with acenaphthene internal standard. This was subsequently extracted with 100ml of Analar grade MIBK (methyl isobutyl ketone). The extract

was allowed to settle, centrifuged and then decanted. This was further eluted with 200ml of 1:3 hexane/dichloromethane to elute the aromatic fraction. The eluate was concentrated over hydrogen and used for analysis by gas chromatography. GC analysis of the sample for BTEX was carried out by a high resolution Phillips Pye Unicam 4500 Gas Chromatograph equipped with a flame ionization detector. A 30m length, 5 μ m film thickness and 0.53mm internal diameter capillary column was used and the peak areas were analyzed with an SRI Model 203 Peak Simple Chromatography Data System. The GC parameters are inlet temperature 220°C split injection, carrier gas helium 10 ml/min constant flow (6.5 psi at 40°C), oven 40°C (3 min) at 7°C/min to 125°C to 250°C (3 min) at 35°C/min, detector FID 300°C, nitrogen makeup gas, 25 ml/min, hydrogen 30 ml/min and air 350 ml/min. 2 μ l of sample was injected. Known weights of Analar grade standards of benzene, toluene, ethyl benzene and o-xylene, spiked with known weight of acenaphthene internal standard was mixed and used for both qualitative and quantitative analyses. 2 μ l of sample was injected. Benzene, toluene, ethyl benzene, o-xylene, m-xylene and p-xylene were determined to obtain the BTEX composition of Activator.

3.4 Quality Assurance Procedure

BEST Project Quality Assurance Plan and BEST Quality Assurance Manual for laboratory standard operating procedures for quality assurance and quality control (QA/QC) were strictly adhered to in the analysis of Activator (Softener Concentrate).

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 Physico-Chemical Parameters

The levels of physico-chemical parameters in Activator (Softener Concentrate) are shown in Table 4.1. The sample phase is a liquid mixture of different chemical composition. The sample was basic with an average pH of 11.70. The sample was colourless. The sample was odourized with perfume. Specific gravity of the sample was 1.001 with reference to water that is 1.000. This showed that the sample is almost of the same specific gravity with water. Activator was miscible with water. The reactivity rating of the sample reflected that it is a stable mixture under normal atmospheric conditions. The evaporating rate with reference to butyl acetate (BuAc = 1) was less than 1. Flammability rate showed that the mixture was not flammable. Boiling point of the sample was approximately 100°C. The freezing point of the sample was almost 0°C. The sulphur content was below detection limit.

Table 4.1: Physico-Chemical Properties of Activator

S/N	Parameter	Result
1	pH	11.70
2	Colour	Colourless
3	Specific Gravity (Water = 1.000)	1.001
4	Phase	Liquid
5	Odour	Perfumed
6	Solubility Rating	Miscible in water
7	Reactivity Rating	Stable
8	Evaporation Rating (BuAc = 1)	< 1
9	Flammability	Not Flammable
10	Boiling Point	~ 100°C
11	Freezing Point	< 0°C
12	Sulphur	< 0.0001ppm

4.2 Heavy Metals

The levels of the heavy metals in the scope of analysis for Activator are shown in Table 4.2. All the heavy metals were below the detection limit.

Table 4.2: Heavy Metal Levels in Activator

S/N	Heavy Metal	Level (ppm)
1	Cadmium (Cd)	< 0.001
2	Iron (Fe)	< 0.001
3	Lead (Pb)	< 0.001
4	Chromium (Cr)	< 0.001
5	Magnesium (Mg)	< 0.001
6	Zinc (Zn)	< 0.001
7	Copper (Cu)	< 0.001
8	Nickel (Ni)	< 0.001
9	Mercury (Hg)	< 0.001
10	Arsenic (As)	< 0.001
11	Cobalt (Co)	< 0.001
12	Vanadium (V)	< 0.001
13	Manganese (Mn)	< 0.001

4.3 Organic Compounds Analysis

The levels of organic compounds in Activator were determined with different methods based on the type of organic compounds. Oil & Grease, Total Hydrocarbons, Polynuclear Aromatic Hydrocarbons, Polychlorinated Biphenyls and BTEX were determined in the sample.

4.3.1 Oil & Grease and Total Hydrocarbons

The levels of oil & grease and total hydrocarbons in Activator are shown in Table 4.3. The oil and grease content of Activator was 0.00036ppm while the total hydrocarbon content of the sample was 0.00024ppm. Chromatogram of the gas chromatographic analysis of Activator is shown in Appendix 4.3.1.

Table 4.3: Oil & Grease and Total Hydrocarbon Levels of Activator

S/N	Parameter	Result (ppm)
1	Oil and Grease	0.00036
2	Total Hydrocarbons	0.00024

4.3.2 Polynuclear Aromatic Hydrocarbons

Table 4.4 shows the correlation coefficient of the selected polynuclear aromatic hydrocarbon standards for naphthalene, phenanthrene, benzo(b)fluoranthene and benzo(g,h,i)perylene. Correlation coefficients of the individual and the average for the selected standards were greater than 0.95, which reflects good calibration for repeatability assurance. The chromatograms of the mixture of standards and calibration curves of the selected polynuclear aromatic standards are shown in Appendix 4.3.2 a – e.

Table 4.4: Correlation Coefficients for Selected Polyaromatic Hydrocarbon Standards

S/N	Polyaromatic Hydrocarbon Standard	Correlation Coefficient
1	Naphthalene	0.99955
2	Phenanthrene	0.99996
3	Benzo(b)fluoranthene	0.99988
4	Benzo(g,h,i)perylene	0.99969

Table 4.5 shows the levels of polynuclear aromatic hydrocarbons in Activator. The total polynuclear aromatic hydrocarbon content of Activator was 0.000895ppb. Naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, benzo(a)anthracene, chrysene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, indeno(1,2,3-cd)pyrene, dibenzo(a,h)anthracene, and benzo(g,h,i)perylene recorded 0.00022, < 0.00001, 0.00001, < 0.00001, 0.00002, 0.00003, 0.00001, 0.00006, 0.00006, 0.00002, 0.00001, 0.00002, 0.00010, 0.00025, 0.00010 and < 0.00001ppb, respectively. The highest and the lowest values were recorded for indeno(1,2,3-cd)pyrene and benzo(g,h,i)perylene, respectively. Chromatogram of the gas chromatographic analysis of Activator is shown in Appendix 4.3.2f.

Table 4.5: Polyaromatic Hydrocarbons in Activator

S/N	Polynuclear Aromatic Hydrocarbon	Result (ppb)
1	Naphthalene	0.00022
2	Acenaphthylene	< 0.00001
3	Acenaphthene	0.00001
4	Fluorene	< 0.00001
5	Phenanthrene	0.00002
6	Anthracene	0.00003
7	Fluoranthene	0.00001
8	Pyrene	0.00006
9	Benzo(a)anthracene	0.00006
10	Chrysene	0.00002
11	Benzo(b)fluoranthene	0.00001
12	Benzo(k)fluoranthene	0.00002
13	Benzo(a)pyrene	0.00010
14	Indeno(1,2,3-cd)pyrene	0.00025
15	Dibenzo(a,h)anthracene	0.00010
16	Benzo(g,h,i)perylene	< 0.00001

4.3.3 Polychlorinated Biphenyls

Table 4.6 shows the correlation coefficients of the selected polychlorinated biphenyl standards for 2,4'-dichlorobiphenyl; 2,2',3,4,5'-pentachlorobiphenyl; 2,2',3,3',4,4',5,6'-octachlorobiphenyl and 2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl. Correlation coefficients of the individual and the average for the selected standards were greater than 0.95, which reflects good calibration for repeatability assurance. The chromatograms of the mixture of standards and calibration curves of the selected polychlorinated biphenyl standards are shown in Appendix 4.3.3 a – e.

Table 4.6: Correlation Coefficients for Selected Polychlorinated Biphenyl Standards

S/N	Polychlorinated Biphenyl Standard	Correlation Coefficient
1	2,4'-dichlorobiphenyl	0.99913
2	2,2',3,4,5'-pentachlorobiphenyl	0.99870
3	2,2',3,3',4,4',5,6-octachlorobiphenyl	0.99938
4	2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	0.99866

Table 4.7 shows the levels of polychlorinated biphenyls in Activator. The total polychlorinated biphenyls in Activator was 0.00117ppb. 2,4'-dichlorobiphenyl; 2,2',5-trichlorobiphenyl; 2,3',3,5'-tetrachlorobiphenyl; 2,2',3,4,5'-pentachlorobiphenyl; 2,2',3,3',4,4'-hexachlorobiphenyl; 2,2',3,3',4,4',5-heptachlorobiphenyl; 2,2',3,3',4,4',5,6-octachlorobiphenyl; 2,2',3,3',4,4',5,5',6-nonachlorobiphenyl and 2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl recorded 0.000008, 0.000040, 0.000104, 0.000302, 0.000132, 0.000195, 0.000194, 0.000098 and 0.000094ppb, respectively. The highest and the lowest values were recorded for 2,2',3,4,5'-pentachlorobiphenyl and 2,4'-dichlorobiphenyl, respectively. Chromatogram of the gas chromatographic analysis of Activator is shown in Appendix 4.3.3f.

Table 4.7: Polychlorinated Biphenyls in Activator

S/N	Polychlorinated Biphenyl	Result (ppb)
1	2,4'-dichlorobiphenyl	0.000008
2	2,2',5-trichlorobiphenyl	0.000040
3	2,3',3,5'-tetrachlorobiphenyl	0.000104
4	2,2',3,4,5'-pentachlorobiphenyl	0.000302
5	2,2',3,3',4,4'-hexachlorobiphenyl	0.000132
6	2,2',3,3',4,4',5-heptachlorobiphenyl	0.000195
7	2,2',3,3',4,4',5,6-octachlorobiphenyl	0.000194
8	2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	0.000098
9	2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	0.000094

4.3.4 BTEX

Table 4.8 shows the correlation coefficients of selected BTEX standards for benzene, toluene, ethyl benzene and o-xylene. Correlation coefficients of the individual and the average for the selected standards were greater than 0.95, which reflects good calibration for repeatability assurance. The chromatogram of the mixture of standards and calibration curves of the selected BTEX standards are shown in Appendix 4.3.4 a – e.

Table 4.8: Correlation Coefficients for Selected BTEX Standards

S/N	BTEX Standard	Correlation Coefficient
1	Benzene	0.99999
2	Toluene	0.99995
3	Ethyl benzene	0.99997
4	o-xylene	0.99982

Table 4.9 shows the levels of BTEX in Activator. The total BTEX in Activator recorded the value of 0.0000143ppb. Benzene, toluene, ethyl benzene, m-xylene, p-xylene and o-xylene recorded < 0.0000001, < 0.0000001, 0.0000022, 0.0000059, 0.0000019 and 0.0000044ppb, respectively. The highest and lowest values were recorded by m-xylene and benzene, respectively. Chromatogram of the gas chromatographic analysis of Activator is shown in Appendix 4.3.4f.

Table 4.9: BTEX in Activator

S/N	BTEX	Result (ppb)
1	Benzene	< 0.0000001
2	Toluene	< 0.0000001
3	Ethyl Benzene	0.0000022
4	o-xylene	0.0000044
5	m-xylene	0.0000059
6	p-xylene	0.0000019

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The results showed the levels of most of the physico-chemical parameters investigated were consistent with the characteristics of Activator (Softener Concentrate). The sulphur content of Activator was quite low. The results showed that all the heavy metals analyzed were below the detection limit and impact on the environment is expected to be insignificant.

The result of oil and grease obtained for Activator was extremely low while the total hydrocarbon level was lower than that of oil and grease. The result agreed with the expectation of the relationship between oil & grease and total hydrocarbons in any medium. The polynuclear aromatic hydrocarbon content in Activator showed that the values were extremely small in parts per billion. These low values cannot exert negative impact on the environment. From naphthalene to benzo(g,h,i)perylene, none of the polynuclear aromatic hydrocarbons was significantly detected. The levels of polychlorinated biphenyl compounds in Activator were in parts per billion. These trace levels of PCBs are not significant and with almost no negative impact on the environment. Finally, the volatile compounds, BTEX, were below detection limit in Activator and have no negative impact on the environment.

Based on the results of this analysis, it can be inferred that any formulated cleaning product must therefore not be too alkaline (pH > 12), not flammable, not lighter than water, not volatile and not reactive. In addition, toxic elements and organic pollutants must be below permissible levels for human use as well as the cleaning equipment and should not have adverse effects on the environment.

In order to further confirm our findings on the concentrations of quality parameters described in this report for Activator, another study may be undertaken to determine its impact on the environment by carrying out aquatic toxicity tests on the sample.

5.2 Recommendation

Extremely low levels or non-detection of the parameters listed in the scope of analysis of solvent cleaning solutions were obtained. The Activator sample is therefore non-toxic and free of pollutant indicators for use in industrial cleaning as:

- No significant amount of total hydrocarbons and oil & grease were obtained;
- Levels of polynuclear aromatic hydrocarbons were in the range of non-detection;
- Polychlorinated biphenyls obtained were in trace amounts; and
- The volatile compounds, BTEX, were low or in the non-detection region.

We therefore recommend that acute aquatic toxicity tests be carried out on samples of Activator (Softener Concentrate) to confirm our findings in this study.

APPENDICES
GAS CHROMATOGRAMS
ACTIVATOR (SOFTENER CONCENTRATE)