# Correlations of Hydrophile-Lipophile Balance with properties affecting water-in-oil emulsion stability of Nigerian crude oils.

R.U. Duru[a,*], L.C. Osuji[b], O.J. Abayeh[b], J.A. Ajienka[c], C.M. Ojinnaka[b]

**ABSTRACT:** Standard methods were used to determine the physical parameters of Nigerian crude oils sampled from Flowing Tubing Head Pressure (FTHP) of well heads in different locations. Direct correlations of the HLB values with the measured physical parameters were not observed. However, with the same $r^2$ value of 87%, the total acid number (TAN) -a precursor to the HLB, increases exponentially as API gravity decreases and increases as the viscosity increases in power regressions. The correlation of the total acid number of the sampled crudes with the dielectric constant values has a coefficient of determination of 0.74 in power regression. The results showed that Nigerian crude oil samples are low in the components that may contribute to the effective HLB and chemical stabilizations. Electrical factors resulting from wax and solid particles may be more responsible for the stability of their emulsions. These serves as a prelude to the application of interrelationships in some crude oil's physical properties in screening and selection of water in-crude oil emulsion demulsifiers. The results also provide a means of predicting the naphthenic acid corrosivity of Nigerian crude oil using their physical parameters.

**Keywords:** Demulsifiers, Dielectric constant, Emulsions, Hydrophile-Lipophile Balance, Screening.

## 1.0 INTRODUCTION

One of the indices that determine the price of crude oil in the international market is the Basic Sediments and Water (BS&W), a term that conveys the quantity of water (and sediments) contained in the crude. Because water in crude oil reduces the holding capacity and causes corrosion of transporting and processing vessels, acceptable values may be as low as 0.5% [1}, [2].

Water is co-produced with crude oil from zero to over fifty percent depending on the age of the well. With the aid of some components of the crude oil such as asphaltenes, resins and fatty acids which act as emulsifiers, a water-in-oil emulsion is formed as the production mixture flows through the facility chokes and valves that provide the required agitation. Sometimes, production techniques such as Enhanced Oil Recovery (EOR) and acidizing or flow enhancement program may require an intentional formation of an emulsion, before subsequent demulsification [3], [4]. Demulsification process has thus become an integral and essential part of crude oil production and processing.

The use of Chemicals has remained the most cost effective means of crude oil demulsification. However, the current bottle test method of screening and selecting an optimum demulsifier concentration is cumbersome and time-consuming [5].

Emulsion stabilization is mainly as a result of interfacial activities of polar fractions of the crude oil – asphaltenes, resins and fatty acids.

The stability of crude oil emulsions depends on many factors and their interrelations. Increase in resin to asphaltene ratio decreases water-in-crude oil emulsion stability due to a reduction in the surface activity of asphaltenes by the formation of resin-solvated aggregates [6]. Temperature, salinity and pH also affect emulsion stability. For example, due to the amphoteric nature of

[a] *World Bank Africa Centre for Excellence, University of Port Harcourt, Nigeria(Research student)*
[b] *Pure & Industrial Chemistry Department, University of Port Harcourt, Nigeria*
[c] *Petroleum & Gas Engineering Department, University of Port Harcourt, Nigeria*
**Corresponding author*

asphaltenes, they acquire positive charge and negative charge respectively at high and low pH environments. At high or low pH, therefore, the molecules become more surface active thereby increasing emulsion stability [7]. Better emulsion resolution has been found to be at pH that is close to neutral [8]. A change in any of these properties due to increased water production will affect one or more of the other properties and may require a change in the demulsifier deployed. Screening and selections that are based on the physical property relationship might be a more efficient means of changing the demulsifier.

HLB is a term used to express the relative simultaneous attraction of a surface active agent for the two phases (usually liquids) that may lead to formation of emulsion. On their properties, the difference (if there is any) between what constitutes an emulsifier and demulsifier of an emulsion system may be only in the HLB of the emulsifier and that of the demulsifier [9]. The surface activity of the major natural surfactants in crude oil (asphaltenes, resins and fatty acids) and indeed most emulsifiers and demulsifiers are polar in characteristics, hence the possible use of HLB and dielectric constants to characterize and rank the demulsifiers against a particular crude oil system.

Since the invention of the HLB system by Griffin in the late 1940's, it has been employed in the formulation and selection of emulsifiers for emulsions [10], [11]. Further studies were made by Shinoda and Saito [12], by comparing it with phase inversion temperature (PIT).

In a study, a linear relationship of the log dielectric constant with the HLB of oils has been found [11]. Ajienka *et. al*., demonstrated the use of dielectric constant to characterize emulsions and demulsifiers by relating them to the emulsion resolution efficiencies [5].

It has been stated that surfactants with low HLB favor water-in-oil emulsions, implying that the natural surfactants in crude oil are lipophilic [9], [10]. Design and formulation of demulsifiers for W/O emulsions (formed by these low HLB surfactants) must, therefore, target high

HLB surfactants [9]. Silva *et. al.* also stated that optimum W/O demulsifier formulation is attained by adding hydrophilic demulsifier to the low lipophilic surfactant to perfectly balance the mixture of interfacial surfactants [13]. The HLB concept has been used to explain the dependence of the affinity of interfacial mixture on the hydrophilicity of the asphaltenes and demulsifier species and their proportions at the interface [14].

From the foregoing, the relationship between HLB and the crude oil properties that have direct bearing on their emulsion stability could serve as the basis for using same to screen and select demulsifiers that will be suitable for a particular emulsion system. The focus of this study is to investigate this relationship using Nigerian crude oil samples and to provide the basis for the use of crude oil-demulsifier physical properties relationship in selecting a demulsifier specifically prepared for the observed physical parameters of a crude oil emulsion.

## 2.0 EXPERIMENTAL

The chemicals and equipment used are listed in Table 1. Crude oil was sampled from flowing tubing head pressure (FTHP) of 13 different well heads in an oil field located in the Niger Delta area of Nigeria with 2-litre plastic cans. The samples, coded as UAB-2, UAE-2, AAA-3, UJE-1, ADO-2, UAE-1, ACO-2, AEG-3, AFA-2, AFI-1, AFI-2, AGJ-2 and AFF-1, were all separately bagged in black polyethylene bags and stored in cartons from the sampling points. The crude oil samples were characterized within ten days from the date of sampling for specific gravity, API gravity, viscosity and base sediments and water (BS&W). The values and methods used are as shown in Table 2.

The API gravity is calculated with formula:

API gravity = (141.5/Dry S.G.) – 131.5

**Table: 1** List of Chemicals and Equipment used.

| S/No. | Chemical | Source |
|---|---|---|
| 1. | n-Heptane (Analar) | BDH |
| 2. | Toluene (ACS) | Hach |
| 3. | Ethanol 96% v/v | BDH |
| 4. | Acetone (Analysis) | Lobie Chemie |
| 5. | Isopropyl alcohol (Analysis | Lobie Chemie |
| 6. | Xylene (Analysis) | Lobie Chemie |
| 7. | Butanone 99.7% | BDH |
| 8. | HCl 37% | Fisher Scientific |
| 9. | Potassium Hydroxide | Fisher Scientific |
| 10. | Potassium Hydrogen Phthalate | BDH |
| | **Equipment** | |
| 1. | KV-8 Viscometer Bath | Stanhope-Seta |
| 3. | Weighing Balance Model ML204/03 | Mettler Toledo |
| 4. | G20 potentiometric titrator/DG116 Probe | Mettler Toledo |
| 5. | BI-870 Dielectric constant meter | BrookHaven |

## 2.1 PROCEDURE FOR COMPARATIVE ASPHALTENE CONTENT IN CRUDE (MODIFIED ASTM D6560)

Each of the samples (10g ± 0.05g) was refluxed with 150 ml n-heptane for 1 hour. The solution was allowed to cool for another 1 hour in a dark fume hood. A dry and pre-weighed filter paper (11cm Whatman # 42) was used to filter the solution. Hot 150 ml n-heptane was used to flush the filter paper further in successive quantities towards the end of the filtration. The filter paper was allowed to dry in the dark for 24 hours after which it was weighed for a number of times till a constant weight was obtained. (The filter paper is handled with forceps after the initial weighing). The dried residue on the filter paper was flushed with hot 100ml toluene to wash out all the asphaltene precipitate and allowed to dry in the fume hood. It was weighed again as above and deducted accordingly to get the weight of asphaltene in the sample. The results are shown in Table 3.

## 2.2 TOTAL ACID NUMBER, SAPONIFICATION NUMBER AND DIELETRIC CONSTANT OF CRUDE OIL SURFACTANTS

Each of the samples was added to a centrifuge tube (100ml) up to the mark followed by 2 drops of demulsifier. The tube's stoppers were loosened slightly and warmed in a water bath maintained at 60 $^{o}$C. After 20 minutes of warming, the tubes were removed and centrifuged for 10 minutes. The separated oil phases were collected with the aid of syringe into labeled containers for the determination of total acid number, saponification number and dielectric constant. The process was repeated for samples with high water content so as to collate up to 60 ml dry oil for the determination of the mentioned parameters.

### 2.2.1 DETERMINATION OF TOTAL ACID NUMBER BY POTENTIOMETRIC TITRATION (ASTM D664)

The dehydrated crude oil samples (20 g ±0.1g) were each used for potentiometric determination of TAN using Mettler Toledo G20 compact titrator with a DG116 probe (for non-aqueous titration). The probe was tested with pH 4 and pH 7 aqueous buffer solutions. Standardization was done with pH 4 and pH 11 aqueous buffer solutions. All other procedures in the ASTM D664 were followed. Total Acid Number was calculated with the formula

TAN (mg KOH/g of sample) = (A - B) x M x 56.1 / W

Where A = Equivalent Point Volume (EQP) of sample, B = EQP volume of Blank, M = Molarity of KOH, W = Weight of the sample. The data and graphical representation of each titration are shown in appendix A.

### 2.2.2 DETERMINATION OF SAPONIFICATION NUMBER BY POTENTIOMETRIC TITRATION (ASTM D94)

The procedure outlined in ASTM D94 was used for the determination of Saponification number by potentiometric titration using Hanna Instrument potentiometer model HI 902. The formula for the calculation of the saponification number is

SAP Number (mg KOH/g of sample) = (B - A) x M x 56.1 / W

Where B= Equivalent Point Volume (EQP) of blank, A = EQP volume of sample, M = Molarity of Hydrochloric acid and W = Weight of sample. The data and graphical representation of each titration are shown in appendix B.

### 2.2.3 CALCULATION OF THE HLB AND MEASUREMENT OF THE DIELECTRIC CONSTANT OF THE CRUDE OIL SAMPLE

The HLB values of the crude oil samples were calculated using the formula:

HLB = 20(1-S/A), [10]

where ‘S' is the saponification number and ‘A' is the acid number. Brookhaven dielectric constant meter model BI-870 was used for to dielectric constant determination. The TAN, SAP, HLB and Dielectric constant values for the crude oil samples and other physical properties are shown in Table 4.

## 3.0 RESULTS AND DISCUSSION

The physical parameters -specific gravity, API gravity and viscosity, exhibited the conventional trends expected in their relationships. Samples with low viscosity for example, showed high values of API gravity and vice versa (Table 2 and Figure 1).

**Table 2:** Measured Physical parameters of samples

| S/No. | Sample | Specific Gr. @ 60 $^{O}$F (ASTM 1298) | API Gravity | Viscosity ($m^2/s$) (ASTM D445) | BS&W (%) (ASTM D4007) |
|---|---|---|---|---|---|
| 1. | AFI-1 | 0.8633 | 32.41 | 4.54 | 14 |
| 2. | AFI-2 | 0.8661 | 31.88 | 3.67 | 25 |
| 3. | UAE-2 | 0.8700 | 31.14 | 6.82 | 16.0 |
| 4. | UAB-2 | 0.8804 | 29.22 | 3.09 | 46.0 |
| 5. | AGJ-2 | 0.9063 | 24.63 | 13.87 | <0.5 |
| 6. | AFF-1 | 0.9088 | 24.20 | 15.59 | - |
| 7. | AAA- | 0.9371 | 19.50 | 58.29 | 14.0 |
| 8. | UJE-1 | 0.9407 | 18.92 | 210.75 | 40.0 |
| 9. | ADO- | 0.9416 | 18.78 | 55.66 | 2.25 |
| 10. | UAE-1 | 0.9446 | 18.30 | 228.65 | 40.0 |
| 11. | AFA-2 | 0.9608 | 15.77 | 305.2 | 28.0 |
| 12. | AEG-3 | 0.9530 | 16.78 | 311.80 | 40.0 |
| 13. | ACO-2 | 0.9675 | 14.75 | 310.49 | 50.0 |

The asphaltene content of crude oil from Niger Delta area of Nigeria is generally low. It is worthy to note that no existing precipitation procedure can give the exact value of the total asphaltene content of crude oil since all asphaltene will not precipitate [15]. The procedure outlined in section 2.1 suffices for comparative estimation of the asphaltene contents in the samples. The values obtained were within the range of values recorded elsewhere [16], [17], [18], [19]

ISSN 2229-5518

and [20]. Generally, the % asphaltene content may not necessarily translate to their extent of participation in the interfacial activities since some will be solvated by the resins to form aggregates. This view is further buttressed by the apparent lack of correlation of the asphaltene content with the HLB values.

**Table 3:** Asphaltene content of Crude oil samples

| S/No. | Well (codified) | A: Weight of dry filter paper + Residue (g) | B: Weight of dry filter paper - Residue (g) | C: Weight of dry Residue (g) = B-C | Comparative estimate of Asphaltene content (%) |
|---|---|---|---|---|---|
| 1 | AFI-1 | 1.161 | 1.0141 | 0.1469 | 1.469 |
| 2 | AFI-2 | 1.1265 | 1.0526 | 0.0739 | 0.739 |
| 3 | UAE-2 | 1.1657 | 1.0731 | 0.0926 | 0.926 |
| 4 | UAB-2 | 1.2903 | 1.2199 | 0.0704 | 0.704 |
| 5 | AGJ-2 | 0.9843 | 0.953 | 0.0313 | 0.313 |
| 6 | AFF-1 | 1.0482 | 1.0004 | 0.0478 | 0.478 |
| 7 | AAA-3 | 1.0956 | 1.0437 | 0.0519 | 0.519 |
| 8 | UJE-1 | 1.1493 | 1.0639 | 0.0854 | 0.854 |
| 9 | ADO-2 | 1.0257 | 0.9836 | 0.0421 | 0.421 |
| 10 | UAE-1 | 1.2224 | 1.09 | 0.1324 | 1.324 |
| 11 | AFA-1 | 1.0826 | 1.0348 | 0.0478 | 0.478 |
| 12 | AEG-3 | 1.213 | 1.1272 | 0.0858 | 0.858 |
| 13 | ACO-2 | 1.3404 | 1.0692 | 0.2712 | 2.712 |

**Table 4**: TAN, SAP Number, HLB and other physical parameters.

| S/No. | Sample | API Gravity | Viscosity ($m^2/s$) (ASTM D445) | BS&W (%) (ASTM D4007) | Dielectric Constant @ 25 $^oC$ (ASTM D924) | Asphaltene content (%) | TAN | SAP Value | HLB |
|---|---|---|---|---|---|---|---|---|---|
| 1. | AFI-1 | 32.41 | 4.54 | 14.0 | 2.39 | 1.5 | 0.10 | 0.04 | 12.0 |
| 2. | AFI-2 | 31.88 | 3.67 | 25.0 | 2.35 | 0.7 | 0.09 | 0.03 | 13.3 |
| 3. | UAE-2 | 31.14 | 6.82 | 16.0 | 2.53 | 0.9 | 0.36 | 0.8 | 13.67 |
| 4. | UAB-2 | 29.22 | 3.09 | 46.0 | 2.38 | 0.7 | 0.16 | 0.09 | 8.75 |
| 5. | AGJ-2 | 24.63 | 13.87 | 42.0 | 2.56 | 0.3 | 0.35 | - | - |
| 6. | AFF-1 | 24.20 | 15.59 | 38.0 | 2.59 | 0.5 | 0.31 | - | - |
| 7. | AAA-3 | 19.50 | 58.29 | 14.0 | 3.04 | 0.5 | 0.52 | 0.22 | 11.58 |
| 8. | UJE-1 | 18.92 | 210.75 | 40.0 | 2.73 | 0.9 | 0.75 | 0.35 | 10.67 |
| 9. | ADO-2 | 18.78 | 55.66 | 2.25 | 2.84 | 0.4 | 1.02 | 0.33 | 13.63 |
| 10. | UAE-1 | 18.30 | 228.65 | 40.0 | 2.74 | 1.3 | 1.07 | 0.57 | 9.25 |
| 11. | AEG-3 | 16.78 | 311.80 | 40.0 | 2.78 | 0.8 | 1.43 | 0.34 | 15.25 |
| 12. | AFA-2 | 15.77 | 305.2 | 28.0 | 2.88 | 1.3 | 1.53 | 0.65 | 11.45 |

ISSN 2229-5518

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 13. | ACO-2 | 14.75 | 310.49 | 50.0 | 2.84 | 2.7 1.16 | 0.35 | 13.98 |

- Not determined due to insufficient sample

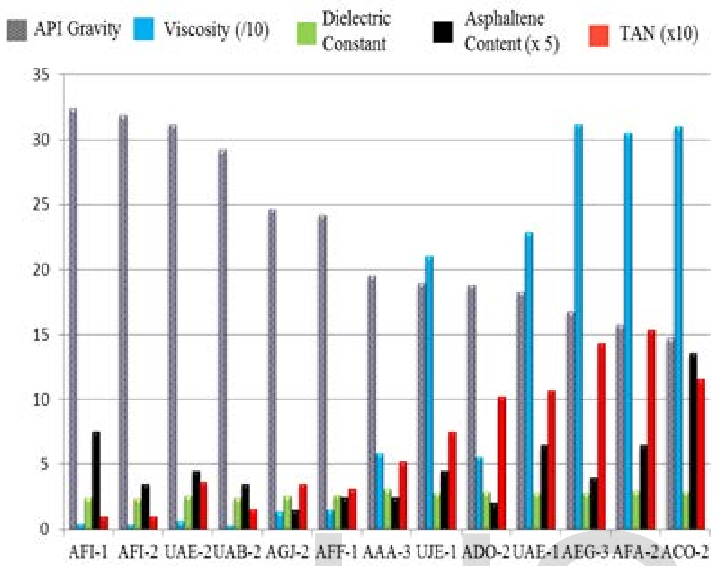



**Figure 1:** Interrelationship of selected physical properties of Nigerian crude oil samples

Extraction partition experiments has been used to show that up to 95% of the acid content of crude oil emulsions are in the oil phase and organic acids are known to be part of resin fraction of the crude oil in general [21]. From Figure 2, it is observed that samples with high API values and low viscosities generally have low total acid number.

R squared values of different regressions for the interrelationships of some of the measured physical properties are shown in Table 5 and the highest values were selected. Specifically, the correlation of the TAN with viscosity and API Gravity is shown in Figures 2 and 3 respectively. It has been noted that crude oil interfacial properties correlate with a carefully measured acid number [22]. With determination coefficients of 87%, it is shown that the API gravity decreases exponentially as the Total acid number increases while viscosity increases as the total acid number increases in power regression with the same $r^2$ value. The correlation of the total acid number of the sampled crudes with the dielectric constant values has coefficient of determination 74% in power regression (Figure 4).

The relationship between the % asphaltene content and the saponification number with the Total Acid Number did not follow any clear pattern. This may be as a result of their interdependence with other properties not captured in the characterization. Consequently, the HLB values did not show any direct correlation with the measured physical parameters.

**Table 5:** Regressions of some of the measured physical properties of samples.

| Parameter | $R^2$ value of Regressions | | | | |
|---|---|---|---|---|---|
| | Exponential | Linear | Logarithmic | Polynomial | Power |
| TAN/API | 0.874 | 0.7929 | 0.8313 | 0.8707 | 0.8711 |
| TAN/ Viscosity | 0.6737 | 0.8239 | 0.8472 | 0.8282 | 0.8746 |
| TAN/ Dielectric constant | 0.7152 | 0.5543 | 0.5723 | 0.6855 | 0.7416 |
| TAN/ % Asphaltene content | 0.06 | 0.1283 | 0.1219 | 0.1307 | 0.0387 |
| TAN/HLB | 0.0533 | 0.0602 | 0.0321 | 0.0764 | 0.0281 |
| Asphaltene/ HLB | 0.0123 | 0.0116 | 2E-05 | 0.1583 | 3E-06 |

ISSN 2229-5518

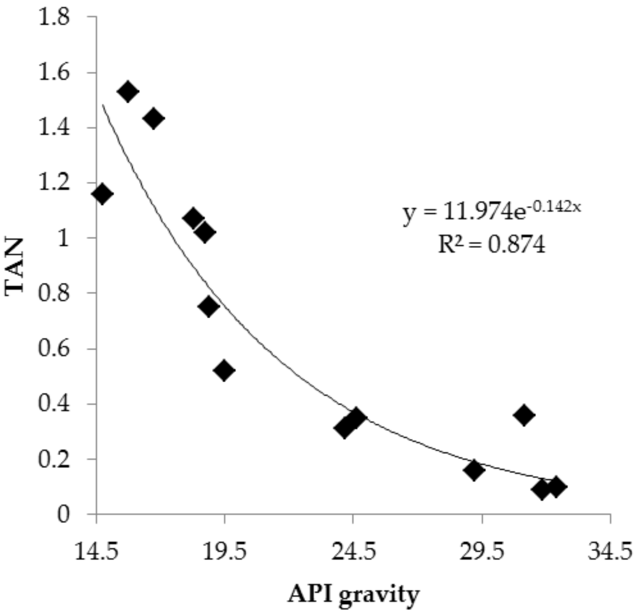



**Figure 2:** Correlation of TAN with API Gravity

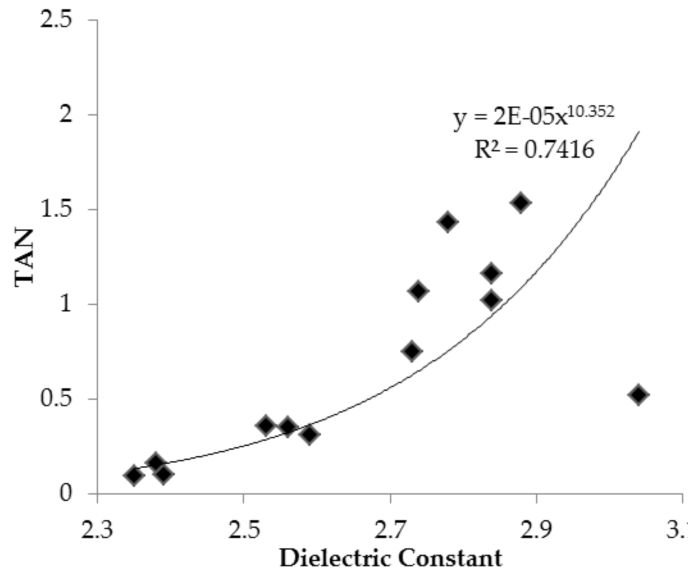



**Figure 4:** Correlation of TAN with Dielectric Constant

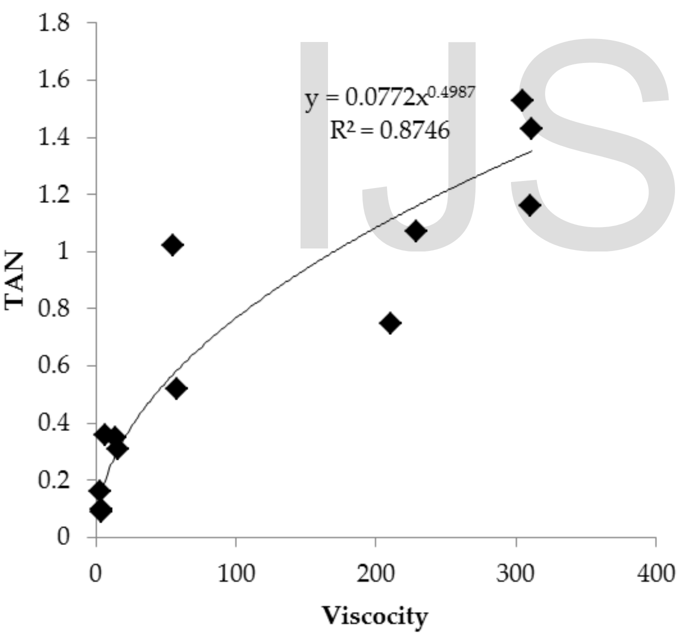



**Figure 3:** Correlation of TAN with Viscosity



**4.0 CONCLUSION**

The results showed good correlations of some of the measured crude oil interfacial properties with the acid number. The little or no correlation observed between HLB and other measured physical properties may be proof that all other interfacially active components of the crude oil such as waxes and fine solid particles other than TAN and asphaltenes contribute to the effective HLB. The calculated HLB values are based on the saponification numbers and total acid numbers alone.

Nigerian crude oil samples are low in asphaltene and fatty acid contents; their emulsion stabilization are more likely to be due to fine sands, solid particles and waxes which give rise to electrostatic stabilizations that are sensitive to dielectric constant. This could further be explained by the apparent lack of correlations of TAN with HLB and asphaltene content. With coefficient of determination of 74% for TAN in relation to the dielectric constant, one can assume that the stabilization of Nigerian crude oil emulsions may be more due to electrical than chemical factors.

Generally, the total contribution of the surface active asphaltenes to the HLB of the Crude oil will depend on other indices like the resin to asphaltene ratio. Since the naphthenic and other fatty acid content of the crude oil are parts of the resin moieties, it may be safe to state that the TAN values of crude oil are dependent on the asphaltenes, the resins and the fatty acids while the HLB depends on the ionic and all other interfacially active components of the crude.

Since the Hydrophile-Lipophile Balance is calculated from total acid number and saponification number values, the observed trend of variation of TAN with some physicochemical properties of crude oil that affects its emulsion stability can serve as the basis for the investigation of HLB application in formulation and screening of chemical demulsifiers. The HLB application will however be more suited to emulsions that are stabilized by chemical factors. This seems realizable when the observed trend of variation of its precursor -TAN with some physicochemical properties of crude oil, which affect emulsion stability is also considered. In doing so however, changes in some other physical parameters such as pH, temperature and electrical stabilization aspects must be accounted for or assumed to be fairly the same for the period of application. A change in pH, for example, will affect the amount of asphaltene that will contribute to the interfacial film since they are ionic entities with basic or acidic group.

The Total acid number, viscosity and dielectric constant values can also be used to predict the Naphthenic acid corrosivity of Nigerian crude oils. From the observed correlations, higher viscosity and dielectric constant of a crude oil would mean an increased Naphthenic acid corrosion. The results can serve as a baseline in the industries for evaluation of corrosion index of production and processing plant parts. At the production stage, however, the knowledge of the salt content of the water component will take precedence.

**Acknowledgement:** For providing the laboratory facilities for this work, the authors are grateful to Production Chemistry and University Liaison Departments of Shell Petroleum Development Company, Port Harcourt and to Fugro Nigeria Limited, Port Harcourt.

## REFERENCES

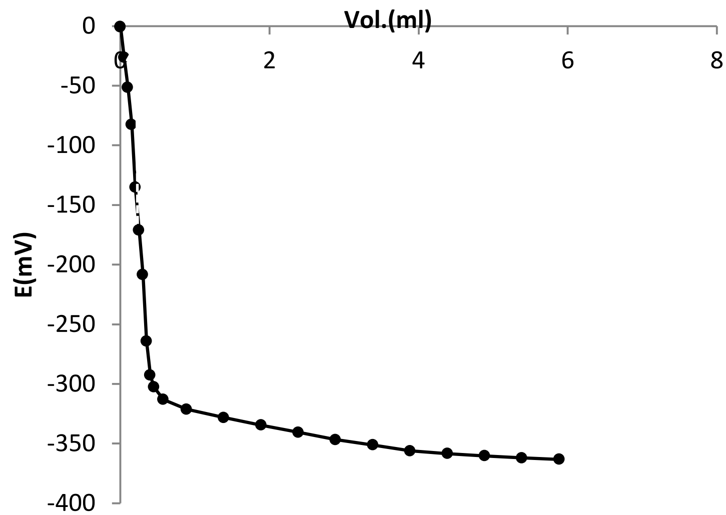
1. J. Sjöblom, N.Aske, I. H. Auflem, Ø. Brandal, T. E. Havre, Sæther, Ø., ... & H. Kallevik, "Our current understanding of water-in-crude oil emulsions.: Recent characterization techniques and high pressure performance", *Advances in Colloid and Interface Science*, *100*, 399-473, 2003.
2. R. Varadaraj, & C. H. Brons, "Demulsification of water-in-oil emulsion" *U.S. Patent No. 8,093,304*, 2012. Washington, DC: U.S. Patent and Trademark Office.
3. C. R McDaniel, K. Kuklenz, &K. Ginsel, (2012)."Composition and method for breaking oil in water emulsions" *U.S. Patent No. 8,168,062, 2012* Washington, DC: U.S. Patent and Trademark Office.
4. S. A. Khoo, "Enhancement of flow in pipeline transportation for crude oil emulsion" *(Doctoral dissertation,* Universiti Malaysia Pahang) 2012.
5. J. A. Ajienka, N. O. Ogbe, and B. C. Ezeaniekwe, "Measurement of dielectric constant of oilfield emulsions and its application to emulsion resolution".*Journal of Petroleum Science and Engineering*, *9*(4), 331-339, 1993.
6. J. D. McLean, & P. K. Kilpatrick, "Effects of asphaltene solvency on stability of water-in-crude-oil emulsions" *Journal of Colloid and Interface Science*, *189*(2), 242-253, 1997.
7. S. Poteau, J. F. Argillier, D. Langevin, F. Pincet, & E. Perez, "Influence of pH on stability and dynamic properties of asphaltenes and other amphiphilic molecules at the oil-water interface" *Energy & Fuels*, *19*(4), 1337-1341, 2005.
8. M. Daaou, & D. Bendedouch, "Water pH and surfactant addition effects on the stability of an Algerian crude oil emulsion" *Journal of Saudi Chemical Society*, *16*(3), 333-337, 2012.
9. C. M.Ojinnaka, J. A. Ajienka, O. J Abayeh, L. C. Osuji & R. U. Duru, "Formulation of best-fit hydrophile/lipophile balance-dielectric permittivity demulsifiers for treatment of crude oil emulsions". *Egyptian Journal of Petroleum*, 2016 http://dx.doi.org/10.1016/j.ejpe.2015.12.005
10. Americas, I.C.I. (1984). The HLB System: A Time-saving Guide to Emulsifier Selection. ICI Americas, Incoporated.
11. R. C. Pasquali, N. Sacco, & C. Bregni, "The studies on hydrophilic–lipophilic balance (HLB): sixty years after William C. Griffin's pioneer work" (1949–2009). *Lat Am J Pharm*, *28*(2), 313-317, 2009.

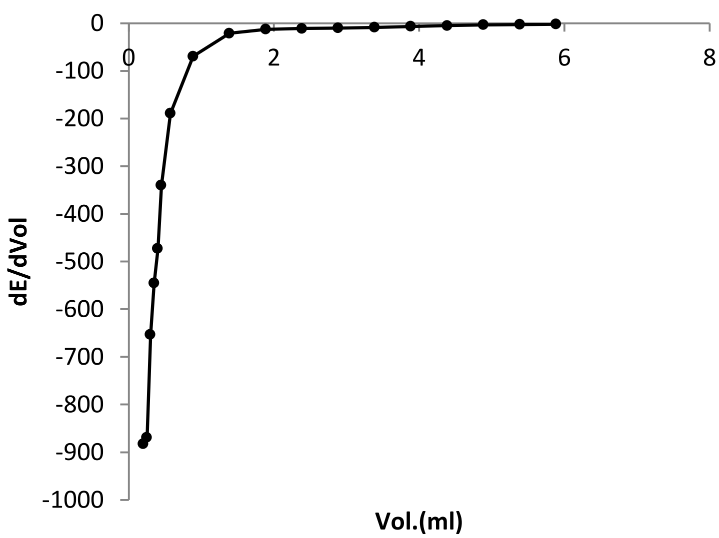
12. K. Shinoda K., & H. Saito "The stability of O/W type emulsions as functions of temperature and the HLB of emulsifiers: the emulsification by PIT-method" *Journal of Colloid and Interface Science*, *30*(2), 258-263, 1969.
13. I. Silva, B. Borges, R. Blanco, M. Rondón, J. L. Salager & J. C. Pereira, "Breaking of water-in-crude oil emulsions. 5. Effect of acid-alkaline additives on the performance of chemical demulsifiers", *Energy & Fuels*, *28*(6), 3587-3593, 2014.
14. J. C Pereira, J. Delgado-Linares, C. Scorzza, M. Rondón, S. Rodríguez & J. L. Salager, "Breaking of Water-in-Crude Oil Emulsions. 4. Estimation of the Demulsifier Surfactant Performance To Destabilize the Asphaltenes Effect" *Energy & Fuels*, *25*(3), 1045-1050, 2011
15. D. Vazquez, & G. A. Mansoori, "Identification and measurement of petroleum precipitates" *Journal of Petroleum Science and Engineering*, *26*(1), 49-55, 2000.
16. E. O. Odebunmi, E. A. Ogunsakin and P. E. P. Ilukhor, "Characterization of crude oil and petroleum products: (1) Elution liquid chromatographic separation and gas chromatographic analysis of crude oils and petroleum product" *Bull. Chem.Soc. Ethiop. 2002*, 16(2), 115-132
17. J. Sjöblom (Ed.). *Emulsions and Emulsion Stability: Surfactant Science Series/61* (Vol. 132). CRC Press, 2005
18. D. Arla, A. Sinquin,T. Palermo, C. Hurtevent, A. Graciaa & C. Dicharry, (2007). "Influence of pH and water content on the type and stability of acidic crude oil emulsions" *Energy & fuels*, *21*(3), 1337-1342, 2007.
19. F. M. Adebiyi & V. Thoss, "Organic and elemental elucidation of asphaltene fraction of Nigerian crude oils" *Fuel*, *118*, 426-431, 2014.
20. N. Aske, "Characterisation of crude oil components, asphaltene aggregation and emulsion stability by means of near infrared spectroscopy and multivariate analysis" *Doctoral dissertation*, Norwegian University of Science and Technology Trondheim), 2002
21. V. Alvarado, X. Wang & M. Moradi, "Stability proxies for water-in-oil emulsions and implications in aqueous-based enhanced oil recovery" *Energies*, *4*(7), 1058-1086, 2011.
22. T. Fan & J. S. Buckley, "Acid number measurements revisited" In *SPE/DOE Symposium on Improved Oil Recovery*. Society of Petroleum Engineers, January 2006.
23. G. C. Laredo, C. R. López, R. E. Alvarez, & J. L. Cano, "Naphthenic acids, total acid number and sulfur content profile characterization in Isthmus and Maya crude oils" *Fuel*, *83*(11), 1689-1695, 2004.
24. American Society for Testing and Materials (ASTM) D1298, D445-15, D4006-11and D4007-11

## Appendix A: Data and graphical representation of potentiometric titrations for TAN determination

### (1) 1st blank

| Vol.(ml) | E(mV) | dE/dVol. |
|---|---|---|
| 0 | -0.5 | NaN |
| 0.001 | -0.8 | NaN |
| 0.051 | -26.2 | NaN |
| 0.101 | -51.4 | NaN |
| 0.151 | -82.5 | NaN |
| 0.201 | -135.2 | -882.58 |
| 0.251 | -171.2 | -869.12 |
| 0.301 | -208.5 | -653.37 |
| 0.351 | -264.2 | -545.52 |
| 0.401 | -292.7 | -473.08 |
| 0.451 | -302.6 | -339.85 |
| 0.576 | -313.1 | -189.16 |
| 0.888 | -321.3 | -69.5 |
| 1.388 | -328.3 | -21.19 |
| 1.888 | -334.5 | -13.02 |
| 2.388 | -340.6 | -11.1 |
| 2.888 | -346.7 | -10.16 |
| 3.388 | -351.2 | -8.88 |
| 3.888 | -356.1 | -6.99 |
| 4.388 | -358.5 | -5.11 |
| 4.888 | -360.3 | -3.66 |
| 5.388 | -362 | -2.76 |
| 5.888 | -363.2 | -2.09 |

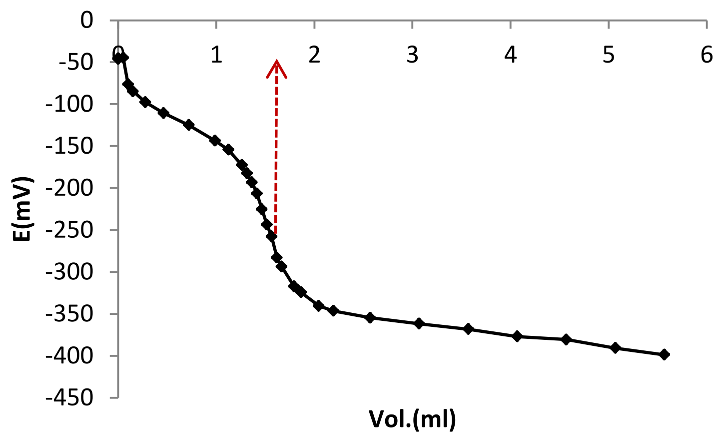


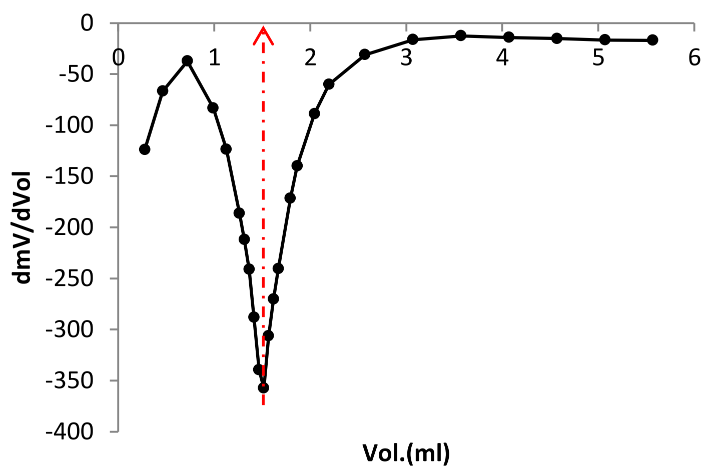




Equivalent Point Volume of Blank:
0.23 ml

**(2) UAE-2**

| Vol.(ml) | E(mV) | dE/dVol. |
|---|---|---|
| 0 | -46.3 | NaN |
| 0.001 | -45.6 | NaN |
| 0.051 | -44.7 | NaN |
| 0.101 | -76.3 | NaN |
| 0.151 | -84.6 | NaN |
| 0.276 | -97.6 | -123.97 |
| 0.463 | -110.6 | -66.77 |
| 0.72 | -124.9 | -37.33 |
| 0.989 | -143.7 | -83.23 |
| 1.125 | -154.4 | -123.61 |
| 1.262 | -172.8 | -186.42 |
| 1.314 | -182.6 | -212.08 |
| 1.364 | -193.2 | -241.3 |
| 1.414 | -206.7 | -288.29 |
| 1.465 | -225.2 | -339.41 |
| 1.515 | -243.5 | -357.42 |
| 1.565 | -257.9 | -306.2 |
| 1.618 | -283.1 | -270.4 |
| 1.668 | -293.7 | -240.58 |
| 1.793 | -317.2 | -171.58 |
| 1.865 | -324.4 | -140 |
| 2.045 | -340.6 | -88.85 |
| 2.195 | -346.5 | -60.21 |
| 2.57 | -354.7 | -31.04 |
| 3.07 | -361.8 | -16.32 |
| 3.57 | -368.2 | -12.64 |
| 4.07 | -377.1 | -14.14 |
| 4.57 | -380.7 | -15.26 |
| 5.07 | -390.8 | -16.72 |
| 5.57 | -398.9 | -16.92 |



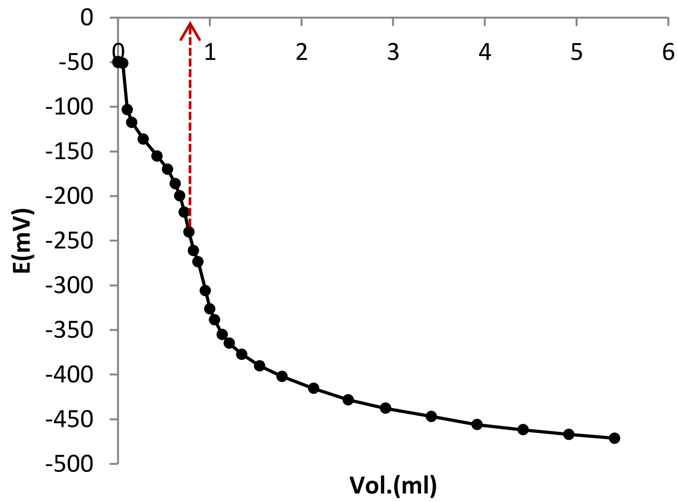



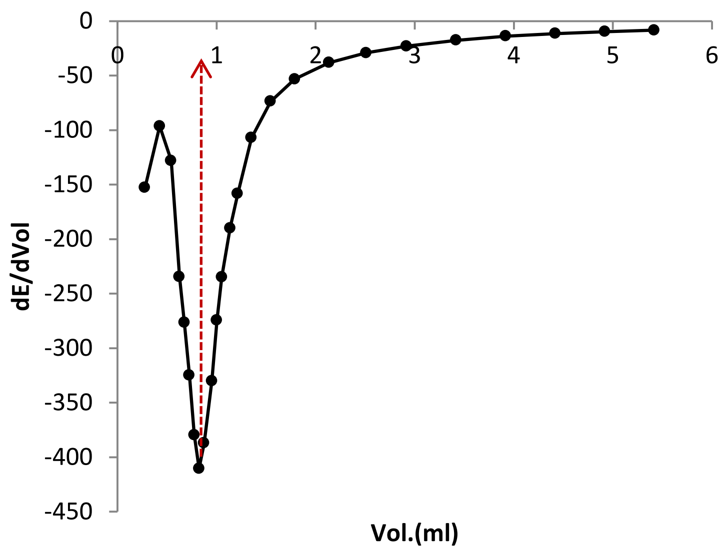

Equivalent point volume for UAE-2: 1.52 ml

TAN for UAE-2 = (1.52 - 0.23) x 0.2805 = 0.36 mgKOH/ g

**(3) UAB-3**

| Vol.(ml) | E(mV) | dE/dVol. |
|---|---|---|
| 0 | -49.4 | NaN |
| 0.001 | -50.5 | NaN |
| 0.051 | -51.2 | NaN |
| 0.101 | -103.3 | NaN |
| 0.151 | -117.5 | NaN |
| 0.276 | -136.4 | -152.83 |
| 0.426 | -155.2 | -96.4 |
| 0.541 | -170.2 | -128.02 |
| 0.624 | -186.2 | -234.67 |
| 0.674 | -199.9 | -276.6 |
| 0.724 | -218 | -324.84 |
| 0.774 | -240.4 | -379.52 |
| 0.824 | -261 | -410.3 |
| 0.874 | -273.6 | -387.03 |
| 0.953 | -306 | -330.07 |
| 1.003 | -326.5 | -274.29 |
| 1.053 | -338.6 | -234.76 |
| 1.137 | -355.1 | -189.86 |
| 1.213 | -365 | -158.39 |
| 1.351 | -377.4 | -107.08 |
| 1.545 | -390.2 | -73.7 |
| 1.789 | -402 | -53.27 |
| 2.135 | -415.5 | -38.23 |
| 2.51 | -428.2 | -29.3 |
| 2.919 | -437.8 | -23.04 |
| 3.419 | -446.9 | -17.6 |
| 3.919 | -456 | -13.86 |
| 4.419 | -461.8 | -11.48 |
| 4.919 | -467 | -9.77 |
| 5.419 | -471.3 | -8.47 |

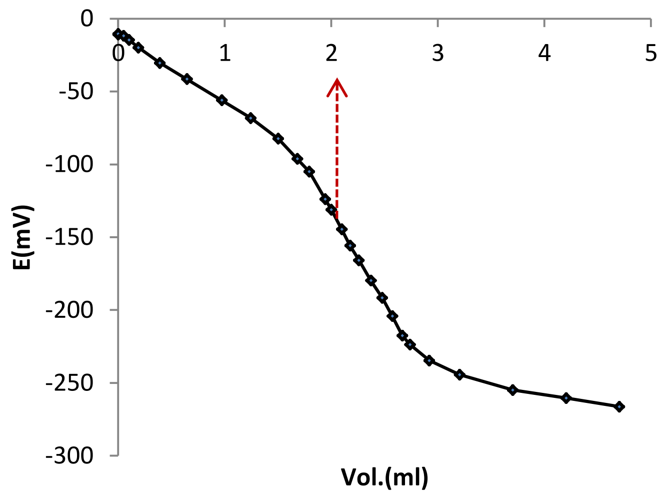


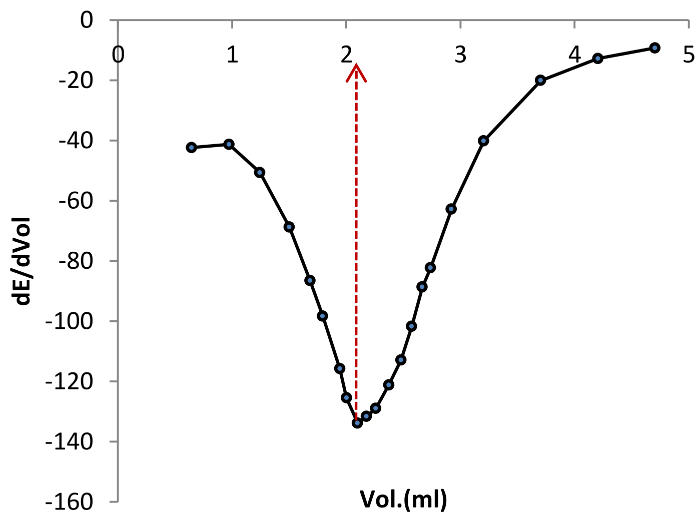




Equivalent point volume for UAB-2: 0.81 ml
TAN for UAB-2 = (0.81 - 0.23) x 0.2805 = 0.16 mgKOH/ g

**(4) AAA-3**

| Vol.(ml) | E(mV) | dE/dVol |
|---|---|---|
| 0 | -11 | NaN |
| 0.001 | -10.6 | NaN |
| 0.051 | -11.9 | NaN |
| 0.101 | -14.7 | NaN |
| 0.19 | -19.9 | NaN |
| 0.391 | -30.5 | NaN |
| 0.645 | -41.6 | -42.37 |
| 0.973 | -56.1 | -41.31 |
| 1.244 | -68.3 | -50.68 |
| 1.502 | -82.5 | -68.8 |
| 1.682 | -96.3 | -86.51 |
| 1.794 | -105.1 | -98.32 |
| 1.943 | -123.9 | -115.74 |
| 2.002 | -131.3 | -125.46 |
| 2.099 | -144.7 | -133.84 |
| 2.177 | -155.9 | -131.65 |
| 2.259 | -165.9 | -128.94 |
| 2.373 | -179.8 | -121.19 |
| 2.479 | -191.7 | -112.92 |
| 2.573 | -204.2 | -101.71 |
| 2.666 | -217.5 | -88.7 |
| 2.739 | -223.7 | -82.25 |
| 2.921 | -234.7 | -62.84 |
| 3.204 | -244.3 | -40.14 |
| 3.704 | -254.9 | -20.09 |
| 4.204 | -260.4 | -12.79 |
| 4.704 | -266.3 | -9.32 |



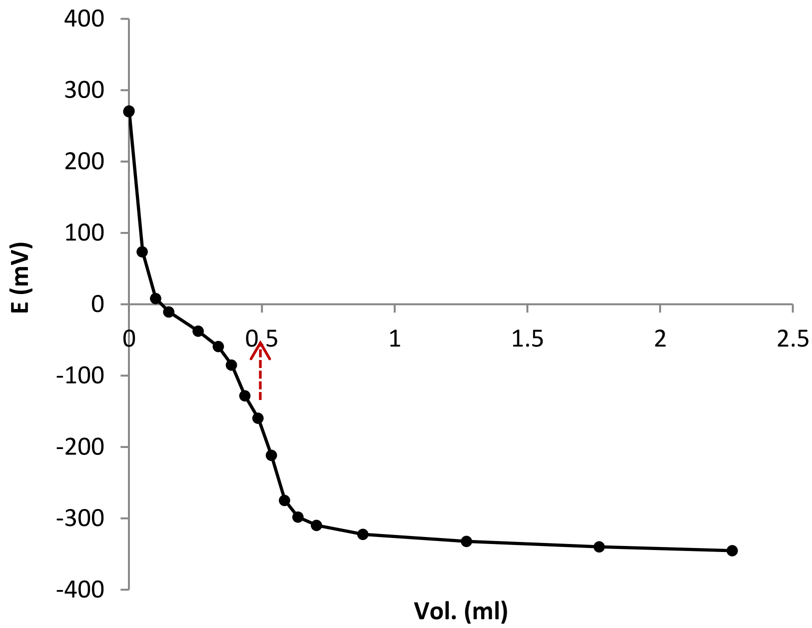



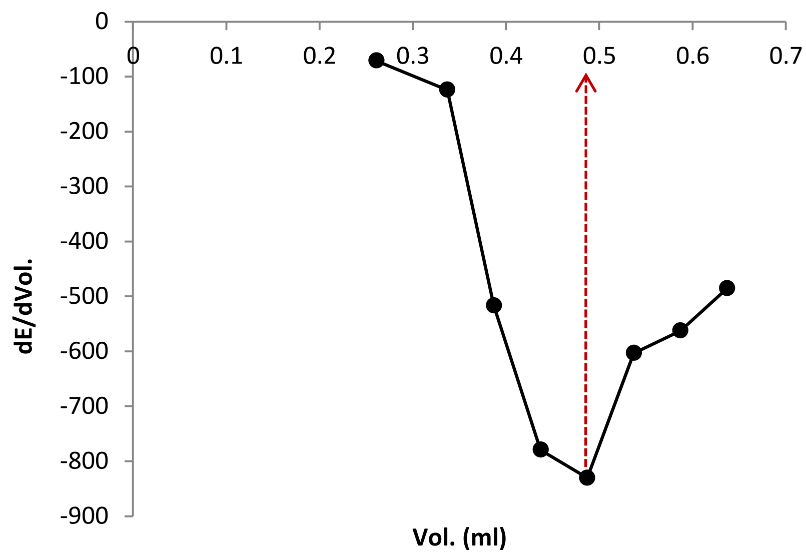

Equivalent point volume for AAA-3: 2.10 ml

TAN for AAA-3 = (2.10 - 0.23) x 0.2805 = 0.52 mgKOH/ g

### (5) 2nd Blank

| Vol.(ml) | E(mV) | dE/dVol. |
|---|---|---|
| 0 | 269.4 | NaN |
| 0.001 | 270.2 | NaN |
| 0.051 | 73.3 | NaN |
| 0.101 | 7.8 | NaN |
| 0.151 | -11 | NaN |
| 0.261 | -37.9 | -71.23 |
| 0.337 | -59.78 | -124.21 |
| 0.387 | -85.5 | -516.91 |
| 0.437 | -128.5 | -779.54 |
| 0.487 | -160.2 | -830.45 |
| 0.537 | -212.1 | -603.47 |
| 0.587 | -275.1 | -562.49 |
| 0.637 | -298.4 | -485.69 |
| 0.707 | -310.2 | NaN |
| 0.882 | -322.8 | NaN |
| 1.272 | -332.6 | NaN |
| 1.772 | -340. 1 | NaN |
| 2.272 | -345.6 | NaN |



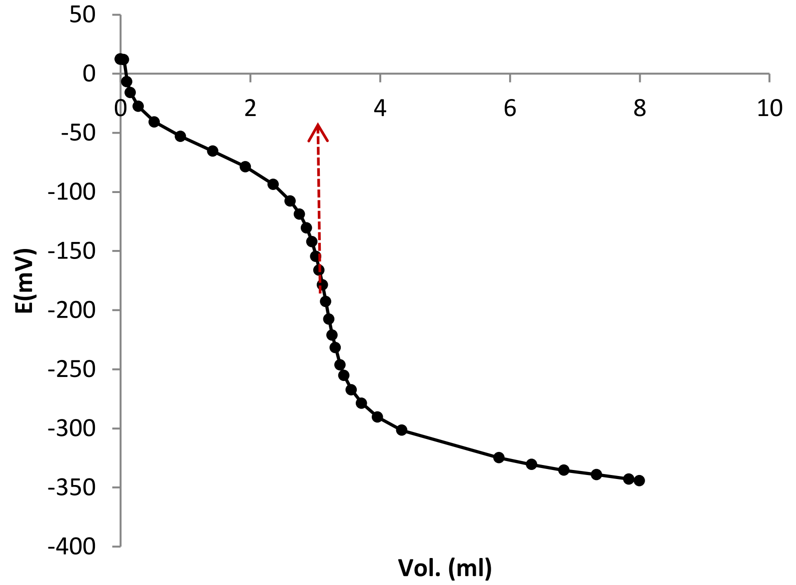



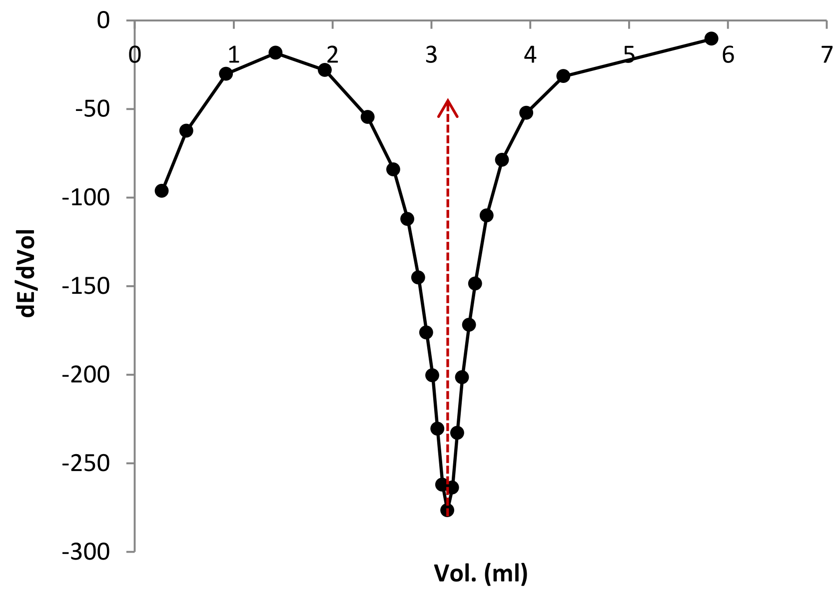

2nd Blank

Equivalent point volume : 0.5ml

**(6) UJE-1**

| Vol.(ml) | E(mV) | dE/dVol. |
|---|---|---|
| 0 | 12.4 | NaN |
| 0.001 | 12 | NaN |
| 0.051 | 11.8 | NaN |
| 0.101 | -6.9 | NaN |
| 0.151 | -16 | NaN |
| 0.276 | -27.8 | -96.43 |
| 0.523 | -40.9 | -62.52 |
| 0.926 | -53.1 | -30.25 |
| 1.426 | -65.6 | -18.41 |
| 1.926 | -78.8 | -28.22 |
| 2.357 | -93.8 | -54.73 |
| 2.618 | -107.9 | -84.25 |
| 2.761 | -118.9 | -112.28 |
| 2.871 | -130.6 | -145.2 |
| 2.952 | -142.3 | -176.22 |
| 3.013 | -154.5 | -200.46 |
| 3.063 | -166.4 | -230.65 |
| 3.113 | -178.8 | -262.12 |
| 3.163 | -192.7 | -276.66 |
| 3.213 | -207.6 | -263.82 |
| 3.263 | -221.2 | -232.89 |
| 3.313 | -231.9 | -201.5 |
| 3.384 | -246.4 | -171.97 |
| 3.445 | -255.3 | -148.64 |
| 3.561 | -267.6 | -110.33 |
| 3.717 | -278.8 | -78.94 |
| 3.963 | -290.6 | -52.26 |
| 4.337 | -301.7 | -31.63 |
| 5.837 | -325 | -10.53 |
| 6.337 | -330.7 | NaN |
| 6.837 | -335.5 | NaN |
| 7.337 | -339.2 | NaN |
| 7.837 | -343 | NaN |
| 8 | -344.4 | NaN |



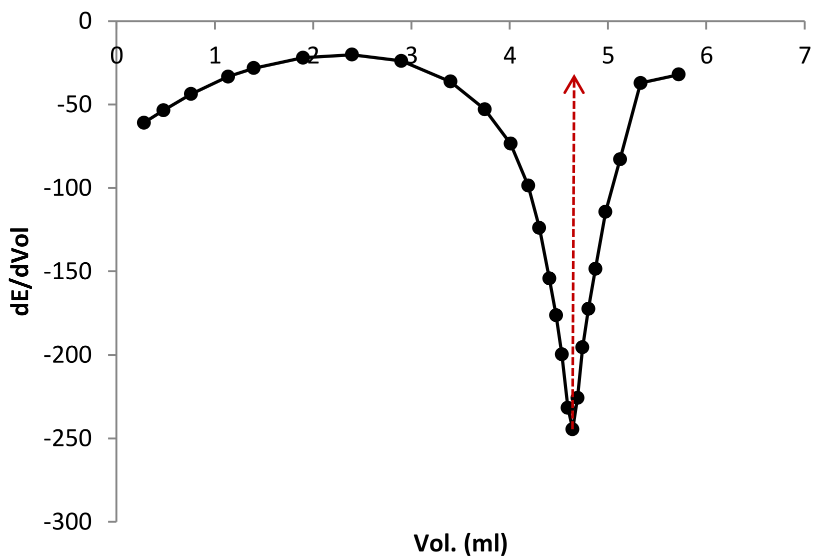



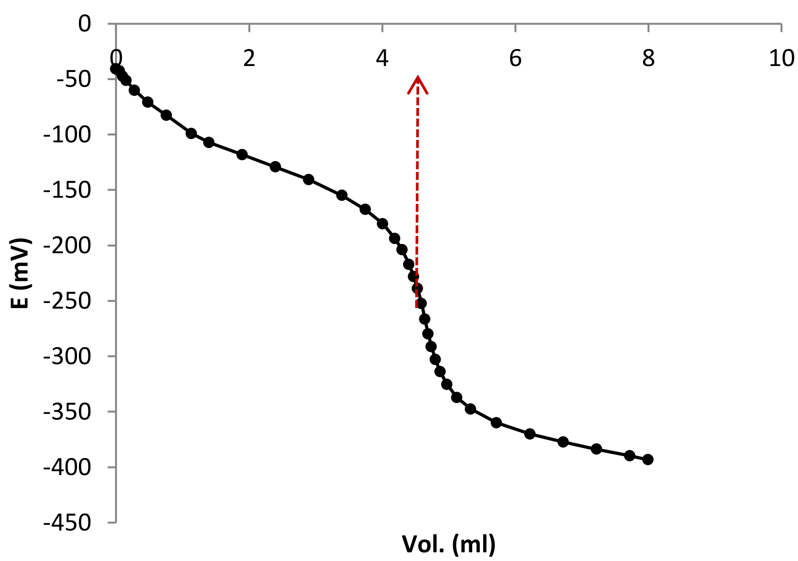

Equivalent Point Volume: 3.17ml

TAN for UJE-1 = (3.17- 0.50) x 0.2805 = 0.75 mgKOH/ g

**(7) ACO-2**

| Vol.(ml) | E(mV) | dE/dVol. |
|---|---|---|
| 0 | -40.9 | NaN |
| 0.001 | -40.9 | NaN |
| 0.051 | -42.8 | NaN |
| 0.101 | -47.7 | NaN |
| 0.152 | -51.5 | NaN |
| 0.28 | -60.3 | -60.96 |
| 0.479 | -71.1 | -53.44 |
| 0.758 | -82.7 | -43.77 |
| 1.136 | -99.2 | -33.32 |
| 1.398 | -107.2 | -28.21 |
| 1.898 | -118.2 | -22.05 |
| 2.398 | -129.3 | -20.19 |
| 2.898 | -140.7 | -23.91 |
| 3.398 | -155 | -36.32 |
| 3.747 | -167.7 | -52.92 |
| 4.01 | -180.6 | -73.55 |
| 4.19 | -193.8 | -98.61 |
| 4.301 | -204 | -123.89 |
| 4.405 | -217.2 | -154.28 |
| 4.473 | -228.3 | -176.27 |
| 4.531 | -238.8 | -199.65 |
| 4.591 | -252.6 | -231.68 |
| 4.641 | -266.5 | -244.52 |
| 4.691 | -279.8 | -225.75 |
| 4.741 | -291.4 | -195.54 |
| 4.801 | -303.1 | -172.52 |
| 4.873 | -314 | -148.41 |
| 4.976 | -325.5 | -114.27 |
| 5.123 | -337.3 | -82.81 |
| 5.332 | -347.6 | -37.17 |
| 5.722 | -360 | -32.13 |
| 6.222 | -370.2 | NaN |
| 6.722 | -377.4 | NaN |
| 7.222 | -383.9 | NaN |
| 7.722 | -389.7 | NaN |
| 8 | -393.4 | NaN |



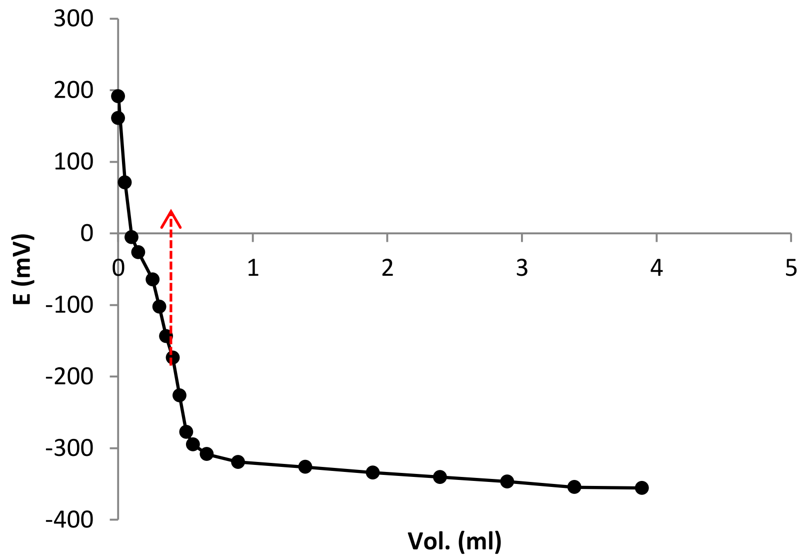

Equivalent Point Volume:
4.63ml

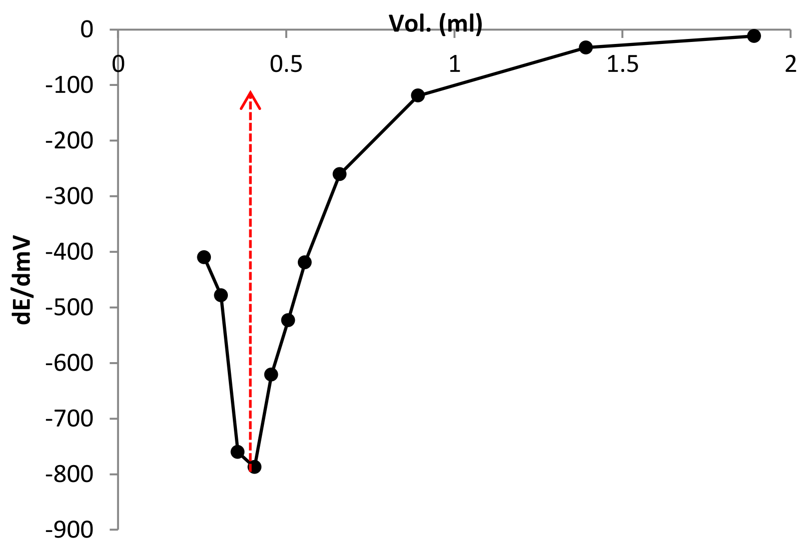
TAN for ACO-2 = (4.63 - 0.50) x 0.2805 = 1.16 mgKOH/g

International Journal of Scientific & Engineering Research, Volume 7, Issue 11, November-2016
ISSN 2229-5518 

**(8) 3rd Blank**

| Vol.(ml) | E(mV) | dE/dVol. |
|---|---|---|
| 0 | 160.7 | NaN |
| 0.001 | 191.4 | NaN |
| 0.051 | 70.9 | NaN |
| 0.101 | -5.6 | NaN |
| 0.151 | -26.6 | NaN |
| 0.256 | -64.5 | -410.06 |
| 0.306 | -102.6 | -478.26 |
| 0.356 | -143.6 | -760.35 |
| 0.406 | -173.9 | -787.47 |
| 0.456 | -226.4 | -621 |
| 0.506 | -277.4 | -523.54 |
| 0.556 | -294.7 | -418.99 |
| 0.659 | -308.4 | -260.56 |
| 0.892 | -319.4 | -119.15 |
| 1.392 | -326.4 | -32.67 |
| 1.892 | -334.2 | -11.99 |
| 2.392 | -340.5 | NaN |
| 2.892 | -346.7 | NaN |
| 3.392 | -354.4 | NaN |
| 3.892 | -355.6 | NaN |



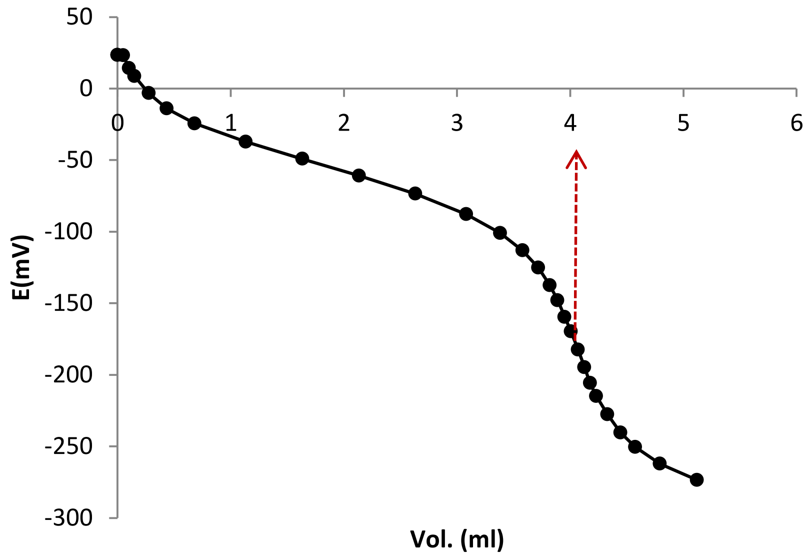



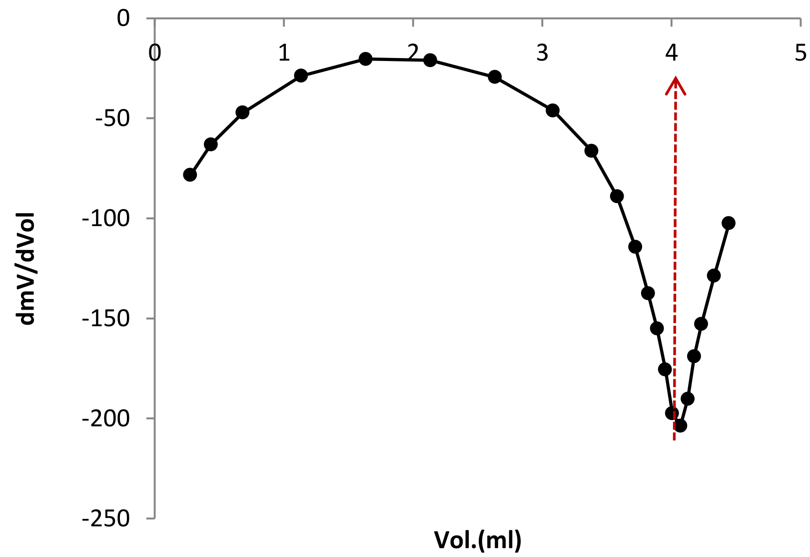

3rd Blank

Equivalent Point Volume: 0.41ml

**(9) ADO-2**

| Vol.(ml) | E(mV) | dE/dVol. |
|---|---|---|
| 0 | 23.3 | NaN |
| 0.001 | 23.4 | NaN |
| 0.051 | 23.2 | NaN |
| 0.101 | 14.3 | NaN |
| 0.151 | 8.5 | NaN |
| 0.276 | -3.3 | -78.4 |
| 0.436 | -14.1 | -63.14 |
| 0.682 | -24.5 | -47.22 |
| 1.134 | -37.2 | -28.78 |
| 1.634 | -49.2 | -20.42 |
| 2.134 | -60.9 | -21.03 |
| 2.634 | -73.6 | -29.48 |
| 3.083 | -87.9 | -46.16 |
| 3.381 | -101 | -66.27 |
| 3.579 | -113.2 | -89.11 |
| 3.72 | -125.3 | -114.35 |
| 3.82 | -137.5 | -137.54 |
| 3.889 | -148 | -155.1 |
| 3.952 | -159.6 | -175.5 |
| 4.006 | -169.7 | -197.46 |
| 4.07 | -182.5 | -203.71 |
| 4.126 | -194.9 | -190.21 |
| 4.176 | -205.8 | -169 |
| 4.232 | -215 | -152.82 |
| 4.33 | -227.8 | -128.67 |
| 4.445 | -240.4 | -102.48 |
| 4.575 | -250.5 | NaN |
| 4.794 | -262.2 | NaN |
| 5.121 | -273.5 | NaN |



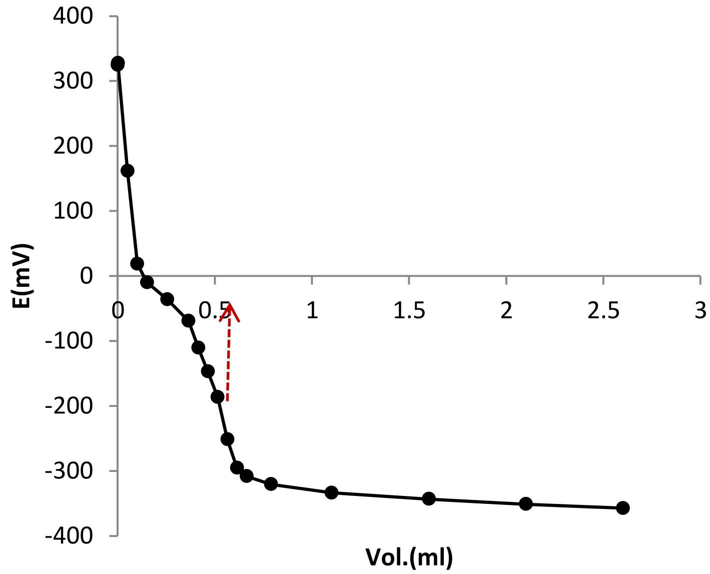

Equivalent point volume: 4.06ml

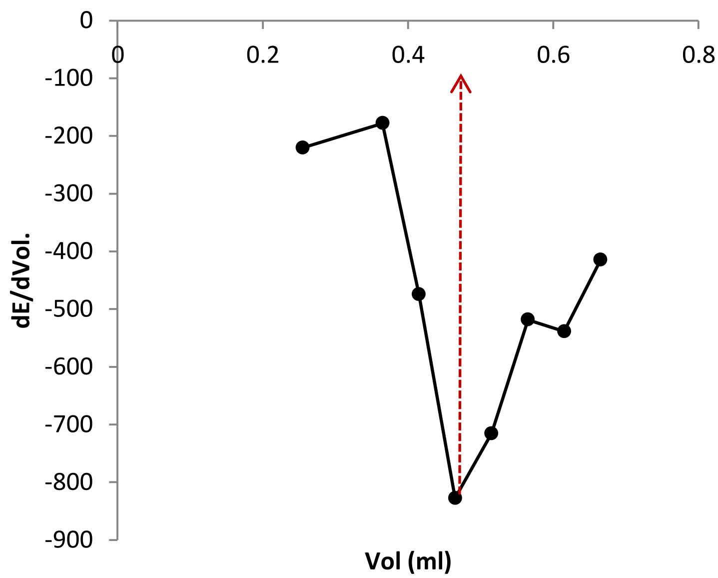
TAN for ADO-2 = (4.06 - 0.41) x 0.2805 = 1.02 mgKOH/ g

International Journal of Scientific & Engineering Research, Volume 7, Issue 11, November-2016
ISSN 2229-5518 

**(10) 4th Blank**

| Vol.(ml) | E(mV) | dE/dVol. |
|---|---|---|
| 0 | 324.8 | NaN |
| 0.001 | 327.6 | NaN |
| 0.051 | 161.5 | NaN |
| 0.101 | 18.5 | NaN |
| 0.151 | -9.9 | NaN |
| 0.255 | -36 | -220.62 |
| 0.365 | -69.4 | -178 |
| 0.415 | -110.5 | -474.33 |
| 0.465 | -147.2 | -827.55 |
| 0.515 | -186.6 | -715.49 |
| 0.565 | -251.6 | -518.53 |
| 0.615 | -295.6 | -538.75 |
| 0.665 | -308.3 | -414.87 |
| 0.79 | -320.6 | NaN |
| 1.102 | -333.8 | NaN |
| 1.602 | -343.4 | NaN |
| 2.102 | -351.3 | NaN |
| 2.602 | -357.3 | NaN |

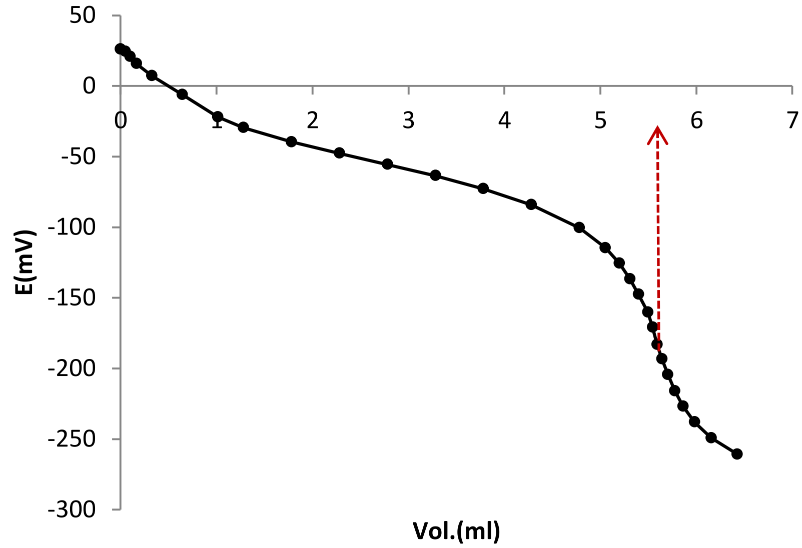


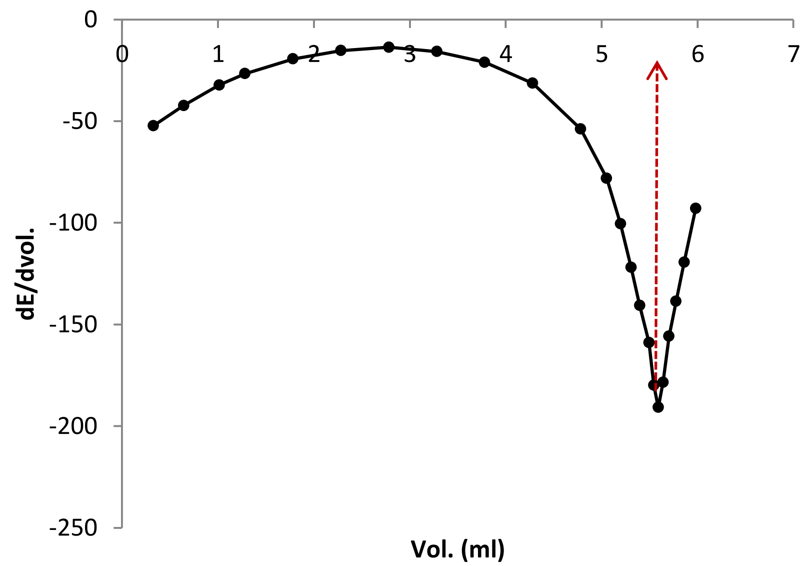




4th Blank Equivalent volume: 0.48ml

**(11) AEG-3**

| Vol(ml) | E(mV) | dE/dVol |
|---|---|---|
| 0 | 26 | NaN |
| 0.001 | 26.1 | NaN |
| 0.051 | 24.5 | NaN |
| 0.101 | 20.7 | NaN |
| 0.167 | 15.7 | NaN |
| 0.328 | 7.1 | -52.24 |
| 0.643 | -6.1 | -42.33 |
| 1.013 | -22 | -32.24 |
| 1.282 | -29.6 | -26.67 |
| 1.782 | -39.6 | -19.42 |
| 2.282 | -47.7 | -15.31 |
| 2.782 | -55.6 | -13.69 |
| 3.282 | -63.6 | -15.8 |
| 3.782 | -72.8 | -21.07 |
| 4.282 | -84.2 | -31.39 |
| 4.782 | -100.5 | -53.91 |
| 5.053 | -114.6 | -78.09 |
| 5.198 | -125.5 | -100.5 |
| 5.308 | -136.6 | -121.91 |
| 5.397 | -147.6 | -140.69 |
| 5.495 | -160.2 | -158.93 |
| 5.544 | -170.9 | -179.87 |
| 5.592 | -183 | -190.79 |
| 5.642 | -193.2 | -178.42 |
| 5.703 | -204.3 | -155.79 |
| 5.776 | -216 | -138.61 |
| 5.861 | -226.8 | -119.33 |
| 5.981 | -238 | -92.86 |
| 6.156 | -249.2 | NaN |
| 6.427 | -260.8 | NaN |



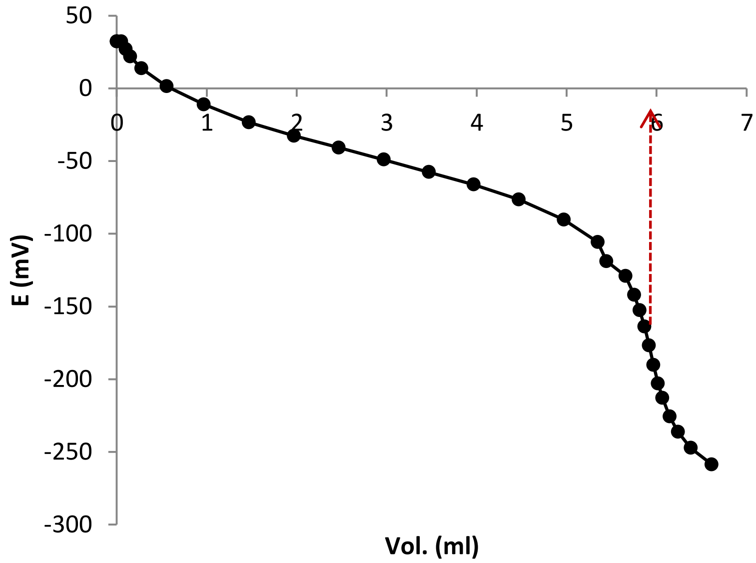



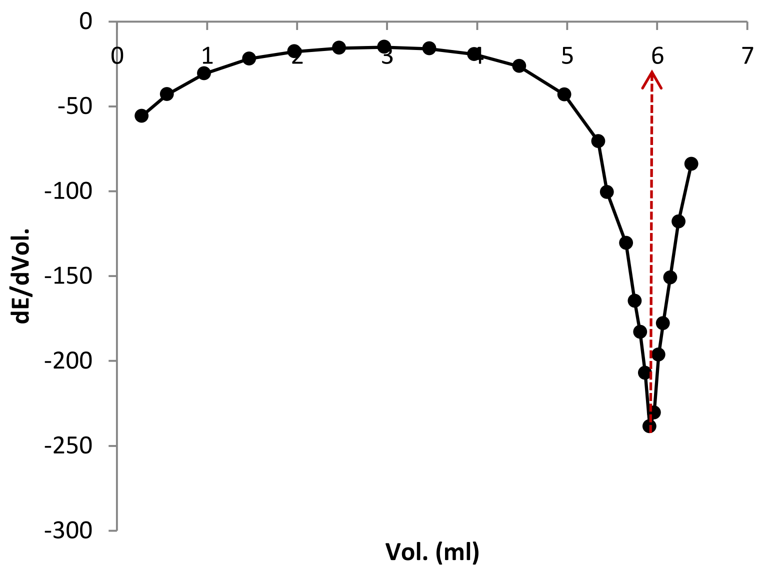

Equivalent Point Volume: 5.58ml

TAN for AEG-3 = (5.58 - 0.48) x 0.2805 = 1.43 mgKOH/g

**(12) AFA-2**

| Vol(ml) | E(mV) | dE/dVol |
|---|---|---|
| 0 | 32.1 | NaN |
| 0.001 | 32.2 | NaN |
| 0.051 | 32.2 | NaN |
| 0.101 | 26.9 | NaN |
| 0.151 | 21.8 | NaN |
| 0.274 | 13.7 | -55.72 |
| 0.556 | 1.3 | -43.05 |
| 0.968 | -11.1 | -30.75 |
| 1.468 | -23.4 | -22.03 |
| 1.968 | -32.7 | -17.72 |
| 2.468 | -40.8 | -15.67 |
| 2.968 | -49.1 | -15.14 |
| 3.468 | -57.6 | -16.04 |
| 3.968 | -66.2 | -19.35 |
| 4.468 | -76.4 | -26.45 |
| 4.968 | -90.3 | -43.1 |
| 5.346 | -105.7 | -70.62 |
| 5.444 | -118.9 | -100.7 |
| 5.654 | -129 | -130.7 |
| 5.751 | -142 | -164.75 |
| 5.812 | -152.6 | -183.13 |
| 5.866 | -163.8 | -207.21 |
| 5.916 | -176.8 | -238.61 |
| 5.966 | -190.3 | -230.5 |
| 6.016 | -203 | -196.53 |
| 6.066 | -212.9 | -177.98 |
| 6.145 | -225.6 | -150.97 |
| 6.237 | -236.2 | -117.97 |
| 6.38 | -247.3 | -84.065 |
| 6.612 | -258.6 | NaN |



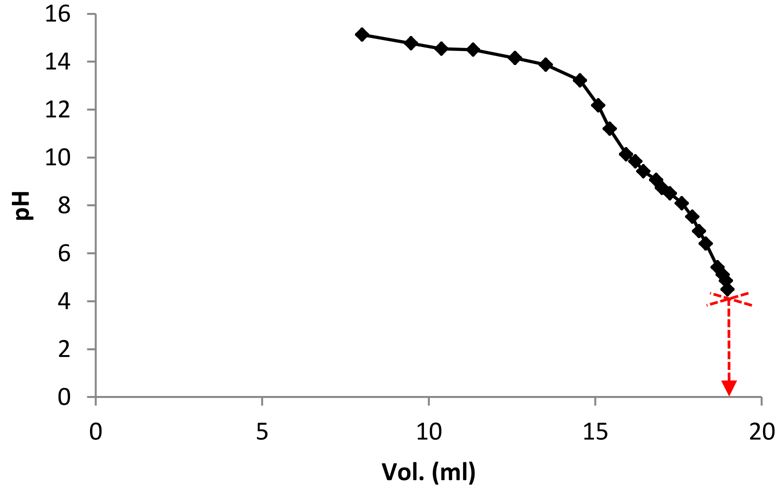

Equivalent point volume: 5.93ml

TAN for AFA-2 = (5.93 - 0.48) x 0.2805 = 1.53 mgKOH/ g

## Appendix B: Data and graphical representation of potentiometric titrations for SAP No. determination

### (1) AAA-3

| Vol.(ml) | pH |
|---|---|
| 8 | 15.13 |
| 9.471 | 14.768 |
| 10.381 | 14.541 |
| 11.333 | 14.503 |
| 12.593 | 14.152 |
| 13.516 | 13.874 |
| 14.546 | 13.221 |
| 15.09 | 12.181 |
| 15.434 | 11.199 |
| 15.931 | 10.143 |
| 16.21 | 9.842 |
| 16.449 | 9.422 |
| 16.832 | 9.065 |
| 17 | 8.733 |
| 17.248 | 8.502 |
| 17.603 | 8.099 |
| 17.916 | 7.526 |
| 18.121 | 6.931 |
| 18.324 | 6.412 |
| 18.674 | 5.427 |
| 18.824 | 5.117 |
| 18.924 | 4.866 |
| 18.973 | 4.5 |



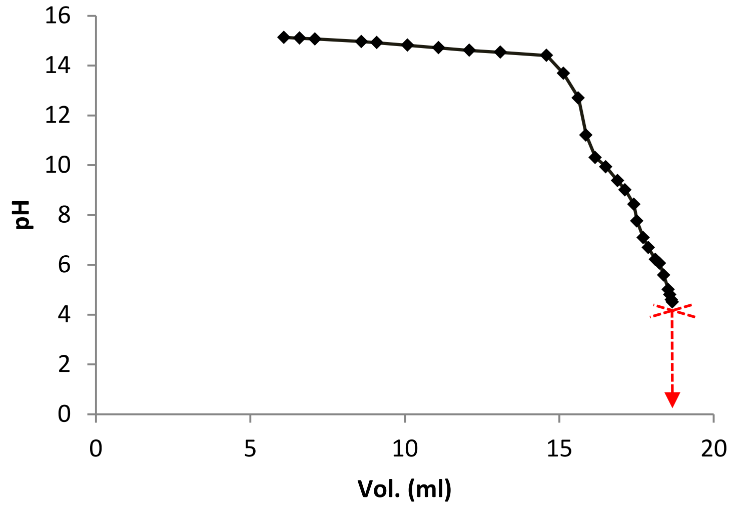

Equivalent point volume: 18.973 ml

Equivalent point volume of blank: 19.192 ml

SAP =56.1 x.113 (19.192-18.073) /20.1307 = 0.219 mgKOH/g

**(2) UAE-2**

| Vol.(ml) | pH |
|---|---|
| 5 | 15.15 |
| 6.096 | 15.127 |
| 6.596 | 15.096 |
| 7.096 | 15.062 |
| 8.596 | 14.964 |
| 9.096 | 14.917 |
| 10.096 | 14.814 |
| 11.096 | 14.712 |
| 12.096 | 14.608 |
| 13.096 | 14.528 |
| 14.596 | 14.403 |
| 15.14 | 13.684 |
| 15.619 | 12.696 |
| 15.869 | 11.209 |
| 16.169 | 10.309 |
| 16.509 | 9.933 |
| 16.892 | 9.376 |
| 17.133 | 9.004 |
| 17.419 | 8.42 |
| 17.519 | 7.755 |
| 17.719 | 7.09 |
| 17.886 | 6.687 |
| 18.113 | 6.215 |
| 18.249 | 6.059 |
| 18.383 | 5.583 |
| 18.533 | 5 |
| 18.583 | 4.802 |
| 18.633 | 4.595 |
| 18.661 | 4.5 |



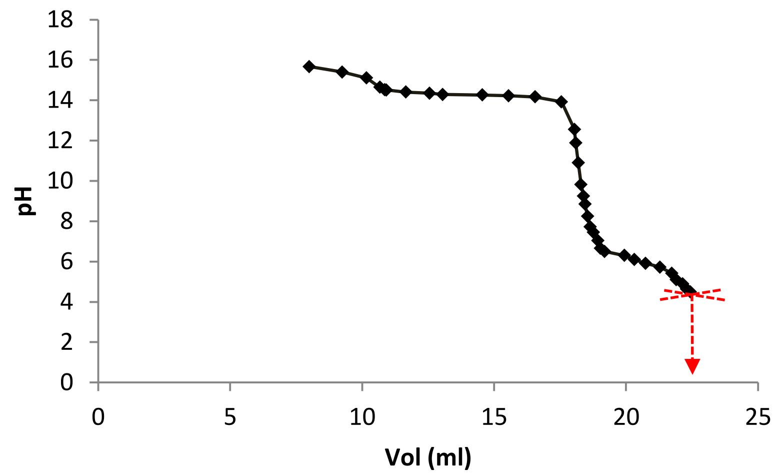

Equivalent point volume: 18.661 ml

Equivalent point volume of blank: 19.192 ml

SAP =56.1 x.113 (19.192-18.661) /20.0443 = 0.80 mgKOH/g

**(3) 2nd Blank**

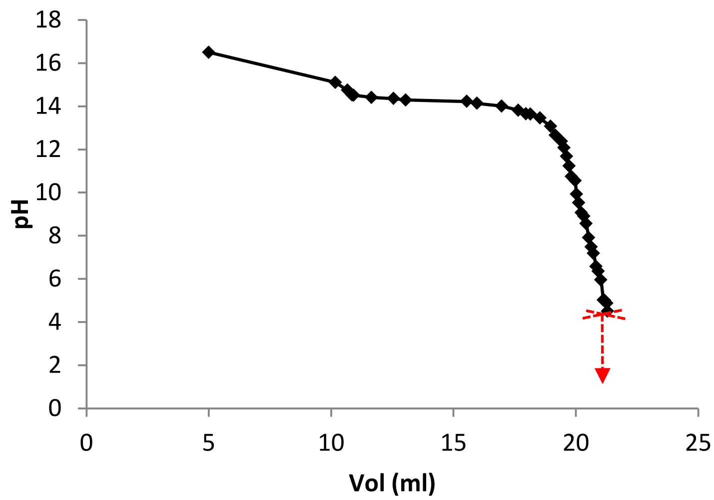
| Vol. (ml) | pH |
|---|---|
| 8 | 15.667 |
| 9.248 | 15.398 |
| 10.174 | 15.101 |
| 10.674 | 14.642 |
| 10.861 | 14.527 |
| 10.922 | 14.512 |
| 11.661 | 14.407 |
| 12.555 | 14.352 |
| 13.055 | 14.289 |
| 14.555 | 14.261 |
| 15.555 | 14.223 |
| 16.555 | 14.168 |
| 17.555 | 13.918 |
| 18.044 | 12.551 |
| 18.094 | 11.884 |
| 18.194 | 10.901 |
| 18.294 | 9.821 |
| 18.394 | 9.238 |
| 18.444 | 8.85 |
| 18.544 | 8.25 |
| 18.644 | 7.715 |
| 18.765 | 7.449 |
| 18.929 | 7.029 |
| 19.036 | 6.649 |
| 19.186 | 6.504 |
| 19.939 | 6.3 |
| 20.321 | 6.093 |
| 20.739 | 5.911 |
| 21.289 | 5.718 |
| 21.729 | 5.421 |
| 21.909 | 5.106 |
| 22.141 | 4.888 |
| 22.287 | 4.63 |
| 22.44 | 4.5 |




Equivalent point volume: 22.440 ml

**(4) UAB-2**

| Vol. (ml) | pH |
|---|---|
| 5 | 16.5 |
| 10.174 | 15.101 |
| 10.674 | 14.742 |
| 10.861 | 14.527 |
| 10.922 | 14.512 |
| 11.661 | 14.407 |
| 12.555 | 14.352 |
| 13.055 | 14.289 |
| 15.555 | 14.223 |
| 15.973 | 14.13 |
| 16.973 | 14.005 |
| 17.659 | 13.816 |
| 17.961 | 13.65 |
| 18.154 | 13.627 |
| 18.539 | 13.448 |
| 18.977 | 13.065 |
| 19.168 | 12.657 |
| 19.412 | 12.37 |
| 19.526 | 12.071 |
| 19.626 | 11.678 |
| 19.726 | 11.225 |
| 19.826 | 10.746 |
| 19.976 | 10.548 |
| 20.026 | 9.925 |
| 20.126 | 9.527 |
| 20.226 | 9.071 |
| 20.326 | 8.907 |
| 20.426 | 8.566 |
| 20.526 | 7.912 |
| 20.626 | 7.471 |
| 20.726 | 7.182 |
| 20.826 | 6.571 |
| 20.926 | 6.344 |
| 21.026 | 5.954 |
| 21.126 | 5.013 |
| 21.176 | 4.995 |
| 21.276 | 4.865 |
| 21.294 | 4.5 |



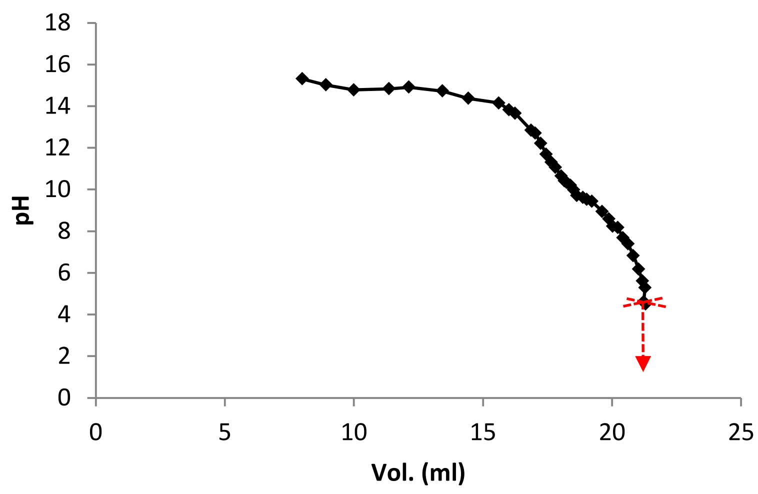

Equivalent point volume: 21.294 ml

Equivalent point volume of blank: 22.440 ml

SAP =56.1 x.113 (22.440-21.294) /20.0194 = 0.36 mgKOH/g

**(5) UJE-2**

| Vol. (ml) | pH |
|---|---|
| 8 | 15.31 |
| 8.922 | 15.014 |
| 9.999 | 14.78 |
| 11.368 | 14.836 |
| 12.136 | 14.909 |
| 13.434 | 14.723 |
| 14.434 | 14.373 |
| 15.62 | 14.152 |
| 16.016 | 13.817 |
| 16.246 | 13.661 |
| 16.866 | 12.841 |
| 17.036 | 12.698 |
| 17.236 | 12.21 |
| 17.454 | 11.691 |
| 17.646 | 11.308 |
| 17.808 | 11.065 |
| 18.034 | 10.64 |
| 18.158 | 10.412 |
| 18.241 | 10.332 |
| 18.391 | 10.194 |
| 18.521 | 9.979 |
| 18.644 | 9.702 |
| 18.874 | 9.616 |
| 19.024 | 9.523 |
| 19.224 | 9.428 |
| 19.624 | 8.943 |
| 19.881 | 8.58 |
| 20.031 | 8.236 |
| 20.224 | 8.165 |
| 20.424 | 7.687 |
| 20.624 | 7.382 |
| 20.829 | 6.822 |
| 21.029 | 6.182 |
| 21.182 | 5.606 |
| 21.282 | 5.291 |
| 21.232 | 4.602 |
| 21.302 | 4.5 |



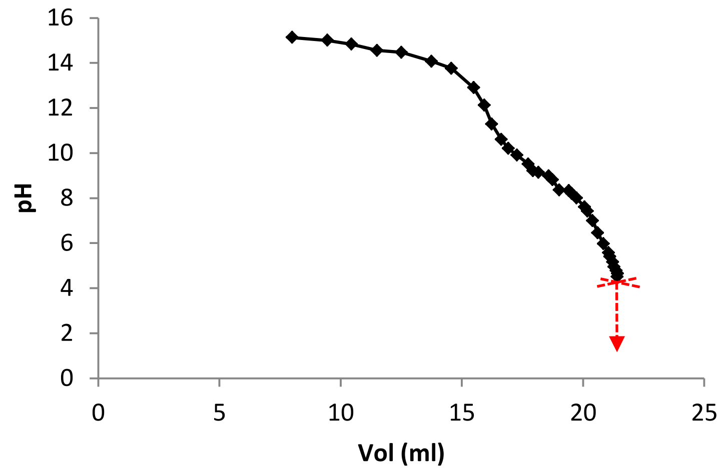

Equivalent point volume: 21.302ml

Equivalent point volume of blank: 22.440 ml

SAP =56.1 x.113 (22.440-21.302) /20.1088 = 0.36 mgKOH/g

**(6) ADO-2**

| Vol. (ml) | pH |
|---|---|
| 8 | 15.13 |
| 9.452 | 14.999 |
| 10.452 | 14.831 |
| 11.509 | 14.552 |
| 12.509 | 14.472 |
| 13.753 | 14.072 |
| 14.573 | 13.758 |
| 15.488 | 12.899 |
| 15.928 | 12.125 |
| 16.233 | 11.286 |
| 16.634 | 10.6 |
| 16.925 | 10.201 |
| 17.278 | 9.904 |
| 17.74 | 9.51 |
| 17.931 | 9.215 |
| 18.158 | 9.139 |
| 18.589 | 8.981 |
| 18.748 | 8.805 |
| 19.014 | 8.36 |
| 19.416 | 8.329 |
| 19.534 | 8.207 |
| 19.728 | 7.999 |
| 20.063 | 7.608 |
| 20.186 | 7.426 |
| 20.399 | 6.998 |
| 20.608 | 6.46 |
| 20.842 | 5.976 |
| 21.048 | 5.575 |
| 21.103 | 5.402 |
| 21.223 | 5.16 |
| 21.273 | 4.942 |
| 21.373 | 4.783 |
| 21.423 | 4.649 |
| 21.409 | 4.5 |



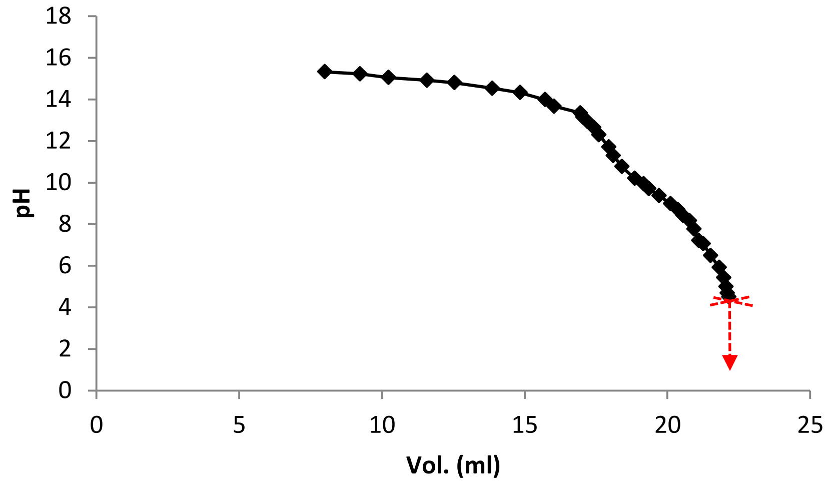

Equivalent point volume: 21.409 ml

Equivalent point volume of blank: 22.440 ml

SAP =56.1 x.113 (22.440-21.409) /20.1066 = 0.33mgKOH/g

**(7) UAB-2**

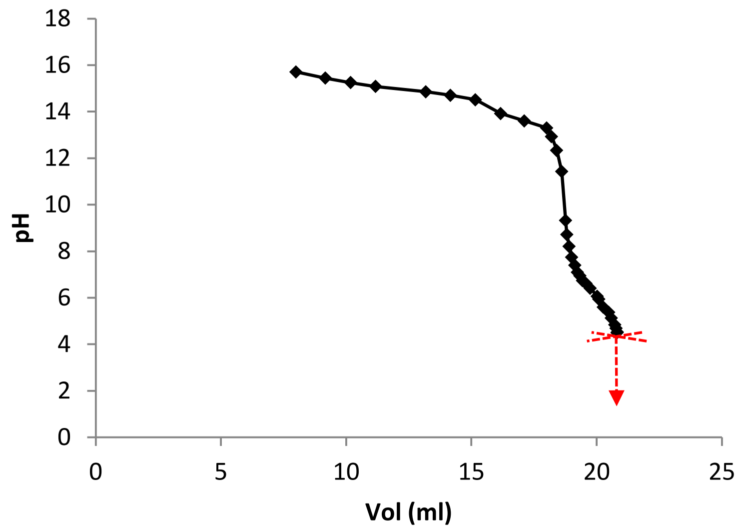
| Vol.(ml) | pH |
|---|---|
| 8 | 15.326 |
| 9.233 | 15.225 |
| 10.233 | 15.052 |
| 11.577 | 14.918 |
| 12.539 | 14.799 |
| 13.867 | 14.53 |
| 14.845 | 14.327 |
| 15.716 | 13.989 |
| 16.026 | 13.667 |
| 16.949 | 13.349 |
| 17.043 | 13.136 |
| 17.202 | 12.945 |
| 17.423 | 12.643 |
| 17.602 | 12.297 |
| 17.948 | 11.703 |
| 18.109 | 11.295 |
| 18.407 | 10.763 |
| 18.853 | 10.2 |
| 19.167 | 9.934 |
| 19.345 | 9.706 |
| 19.712 | 9.363 |
| 20.114 | 8.982 |
| 20.366 | 8.687 |
| 20.534 | 8.427 |
| 20.778 | 8.166 |
| 20.929 | 7.76 |
| 21.098 | 7.207 |
| 21.26 | 7.058 |
| 21.51 | 6.492 |
| 21.815 | 5.921 |
| 21.98 | 5.423 |
| 22.051 | 4.987 |
| 22.1 | 4.691 |
| 22.151 | 4.5 |




Equivalent point volume: 22.151 ml
Equivalent Point Volume of blank: 22.440 ml
SAP =56.1 x.113 (22.440-22.151)/20.0472
= 0.091 mgKOH/g

**(8) 3rd Blank**

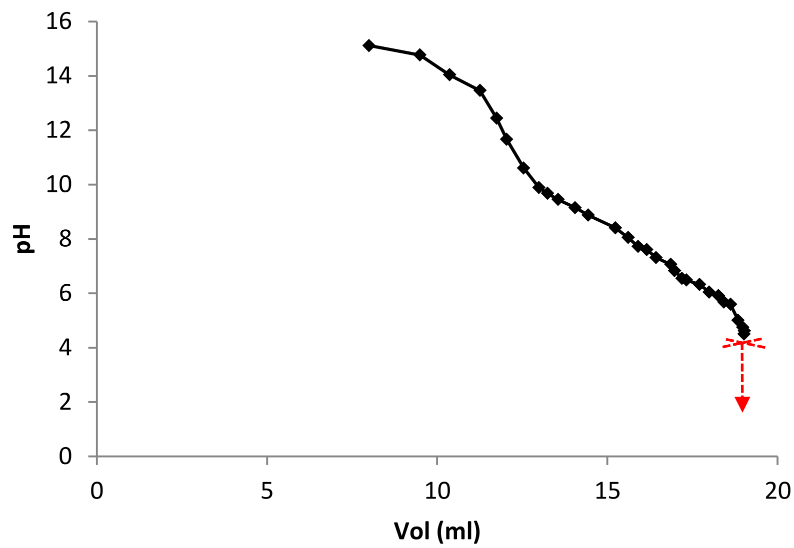
| Vol.(ml) | pH |
|---|---|
| 15.71 | 15.107 |
| 15.44 | 14.76 |
| 15.247 | 14.032 |
| 15.081 | 13.456 |
| 14.852 | 12.44 |
| 14.7 | 11.661 |
| 14.508 | 10.606 |
| 13.918 | 9.884 |
| 13.602 | 9.678 |
| 13.298 | 9.446 |
| 12.922 | 9.151 |
| 12.328 | 8.871 |
| 11.423 | 8.407 |
| 9.327 | 8.051 |
| 8.714 | 7.717 |
| 8.209 | 7.608 |
| 7.745 | 7.314 |
| 7.401 | 7.061 |
| 7.094 | 6.83 |
| 6.935 | 6.547 |
| 6.735 | 6.484 |
| 6.606 | 6.318 |
| 6.4 | 6.045 |
| 6.061 | 5.911 |
| 5.944 | 5.681 |
| 5.594 | 5.594 |
| 5.375 | 5.009 |
| 5.121 | 4.749 |
| 4.831 | 4.617 |
| 4.68 | 4.5 |




Equivalent point volume:
20.824 ml

**(9) UAE-1**

| Vol. (ml) | pH |
|---|---|
| 8 | 15.107 |
| 9.495 | 14.76 |
| 10.363 | 14.032 |
| 11.257 | 13.456 |
| 11.746 | 12.44 |
| 12.043 | 11.661 |
| 12.541 | 10.606 |
| 12.991 | 9.884 |
| 13.241 | 9.678 |
| 13.553 | 9.446 |
| 14.054 | 9.151 |
| 14.438 | 8.871 |
| 15.236 | 8.407 |
| 15.61 | 8.051 |
| 15.904 | 7.717 |
| 16.156 | 7.608 |
| 16.429 | 7.314 |
| 16.858 | 7.061 |
| 16.975 | 6.83 |
| 17.193 | 6.547 |
| 17.327 | 6.484 |
| 17.711 | 6.318 |
| 17.993 | 6.045 |
| 18.266 | 5.911 |
| 18.42 | 5.681 |
| 18.616 | 5.594 |
| 18.839 | 5.009 |
| 18.974 | 4.749 |
| 19.024 | 4.617 |
| 19.019 | 4.5 |



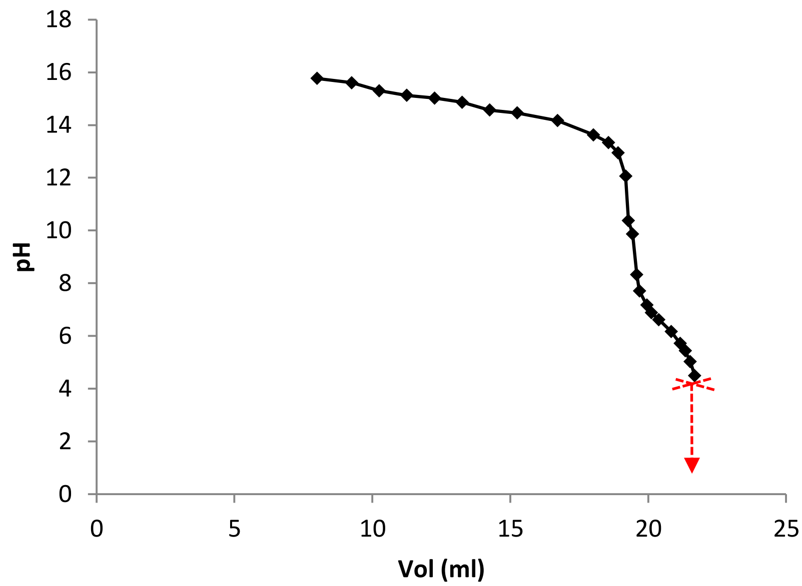

Equivalent point volume: 19.019 ml

Equivalent point volume: 20.824 ml

SAP =56.1 x.113 (20.824-19.019) /20.0428 = 0.57 mgKOH/ g

**(10) 4th Blank**

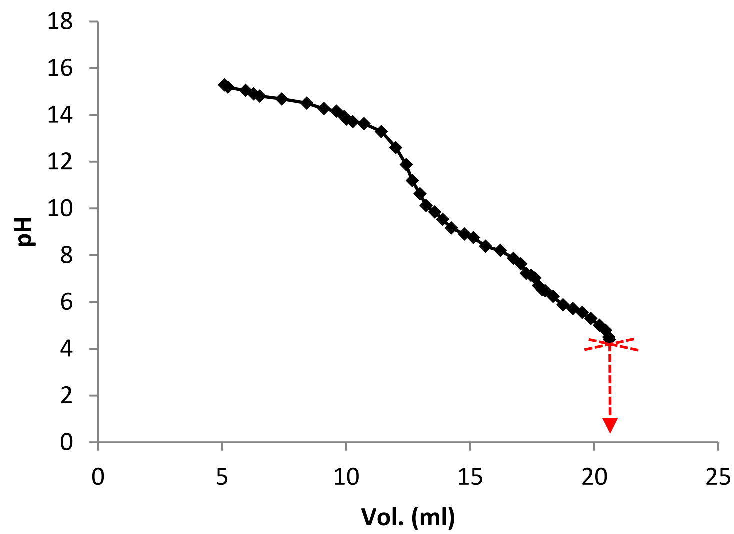
| Vol. (ml) | pH |
|---|---|
| 8 | 15.764 |
| 9.25 | 15.604 |
| 10.25 | 15.303 |
| 11.25 | 15.122 |
| 12.25 | 15.021 |
| 13.25 | 14.862 |
| 14.25 | 14.566 |
| 15.25 | 14.455 |
| 16.716 | 14.164 |
| 18.018 | 13.626 |
| 18.557 | 13.338 |
| 18.911 | 12.95 |
| 19.183 | 12.06 |
| 19.283 | 10.373 |
| 19.433 | 9.868 |
| 19.583 | 8.33 |
| 19.683 | 7.712 |
| 19.949 | 7.17 |
| 20.119 | 6.88 |
| 20.384 | 6.619 |
| 20.829 | 6.171 |
| 21.156 | 5.725 |
| 21.343 | 5.439 |
| 21.523 | 5.03 |
| 21.682 | 4.5 |




Equivalent point volume: 21.682 ml

**(11) AEG-3**

| Vol. (ml) | pH |
|---|---|
| 5.1 | 15.273 |
| 5.25 | 15.177 |
| 5.962 | 15.038 |
| 6.274 | 14.9 |
| 6.528 | 14.8 |
| 7.418 | 14.675 |
| 8.418 | 14.5 |
| 9.114 | 14.262 |
| 9.618 | 14.16 |
| 9.923 | 13.922 |
| 10.023 | 13.812 |
| 10.277 | 13.701 |
| 10.725 | 13.623 |
| 11.425 | 13.28 |
| 12.006 | 12.6 |
| 12.425 | 11.863 |
| 12.673 | 11.19 |
| 12.983 | 10.621 |
| 13.233 | 10.116 |
| 13.579 | 9.85 |
| 13.897 | 9.528 |
| 14.249 | 9.161 |
| 14.78 | 8.899 |
| 15.144 | 8.747 |
| 15.634 | 8.379 |
| 16.218 | 8.202 |
| 16.757 | 7.866 |
| 17.052 | 7.626 |
| 17.269 | 7.213 |
| 17.461 | 7.13 |
| 17.613 | 7.023 |
| 17.757 | 6.69 |
| 17.912 | 6.519 |
| 18.034 | 6.478 |
| 18.349 | 6.23 |
| 18.748 | 5.875 |
| 19.148 | 5.705 |
| 19.521 | 5.549 |
| 19.87 | 5.283 |
| 20.222 | 4.994 |
| 20.471 | 4.785 |
| 20.591 | 4.5 |
| 20.621 | 4.388 |



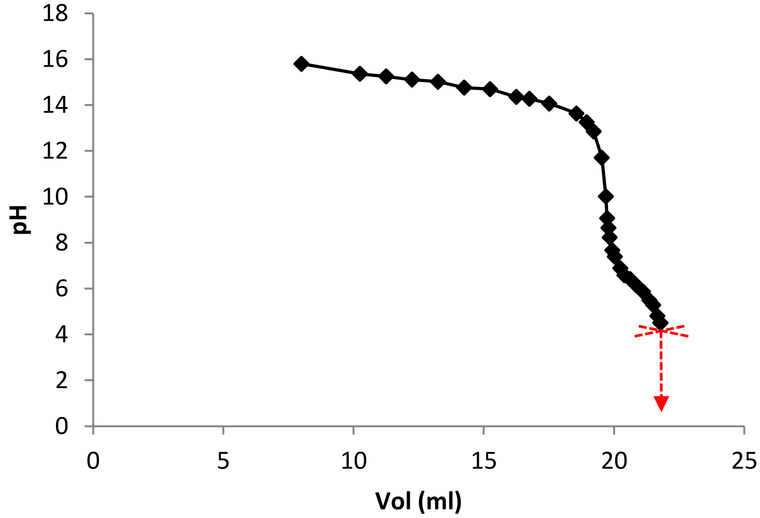

Equivalent point volume: 20.607ml

Equivalent point volume of blank: 21.682 ml

SAP =56.1 x.113 (21.682-20.607) /20.0702 = 0.34 mgKOH/ g

**(12) 5th Blank**

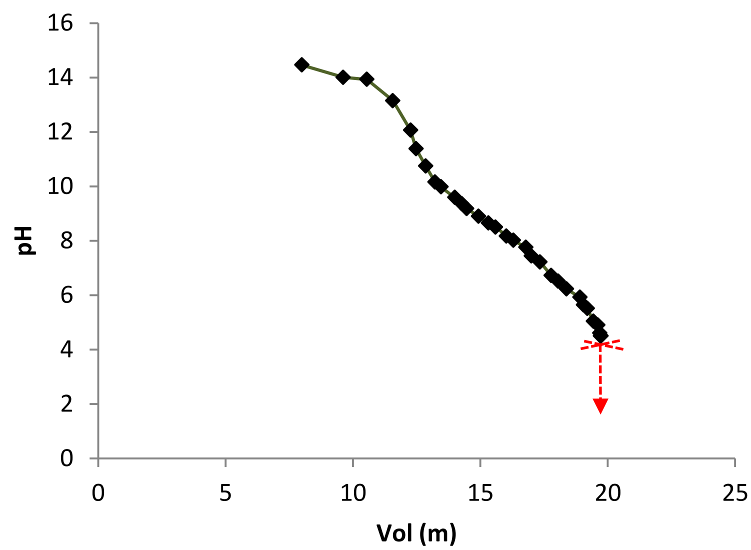
| Vol. (ml) | pH |
|---|---|
| 8 | 15.795 |
| 10.25 | 15.355 |
| 11.25 | 15.243 |
| 12.25 | 15.107 |
| 13.25 | 15.018 |
| 14.25 | 14.76 |
| 15.25 | 14.691 |
| 16.25 | 14.352 |
| 16.75 | 14.266 |
| 17.515 | 14.065 |
| 18.561 | 13.635 |
| 18.953 | 13.25 |
| 19.22 | 12.842 |
| 19.536 | 11.698 |
| 19.686 | 10.015 |
| 19.736 | 9.062 |
| 19.786 | 8.645 |
| 19.836 | 8.231 |
| 19.936 | 7.671 |
| 20.036 | 7.392 |
| 20.239 | 6.885 |
| 20.405 | 6.585 |
| 20.629 | 6.411 |
| 20.866 | 6.132 |
| 21.116 | 5.871 |
| 21.365 | 5.492 |
| 21.51 | 5.285 |
| 21.663 | 4.799 |
| 21.778 | 4.512 |
| 21.786 | 4.5 |




Equivalent point volume: 21.778 ml

**(13) AFA-2**

| Vol. (ml) | pH |
|---|---|
| 8 | 14.458 |
| 9.621 | 14.002 |
| 10.553 | 13.926 |
| 11.571 | 13.146 |
| 12.268 | 12.058 |
| 12.478 | 11.38 |
| 12.853 | 10.748 |
| 13.226 | 10.152 |
| 13.456 | 9.975 |
| 13.997 | 9.59 |
| 14.281 | 9.36 |
| 14.472 | 9.175 |
| 14.929 | 8.903 |
| 15.317 | 8.646 |
| 15.598 | 8.502 |
| 16.029 | 8.172 |
| 16.3 | 8.013 |
| 16.79 | 7.759 |
| 17.001 | 7.436 |
| 17.343 | 7.222 |
| 17.778 | 6.726 |
| 18.056 | 6.51 |
| 18.388 | 6.226 |
| 18.915 | 5.919 |
| 19.049 | 5.64 |
| 19.206 | 5.511 |
| 19.44 | 5.038 |
| 19.618 | 4.905 |
| 19.686 | 4.601 |
| 19.736 | 4.503 |
| 19.711 | 4.5 |



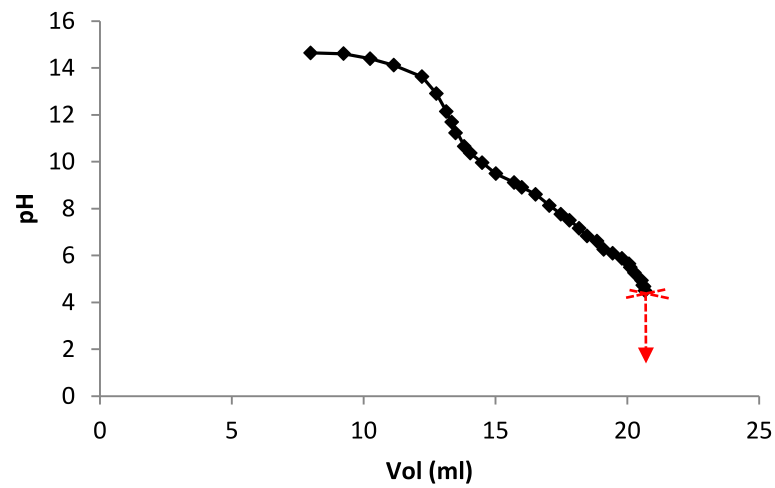

Equivalent point volume: 19.711 ml

Equivalent point volume of blank: 21.778 ml

SAP =56.1 x.113 (21.778 - 19.711) /20.0378 = 0.65 mgKOH/ g

**(14) ACO-2**

| Vol. (ml) | pH |
|---|---|
| 8 | 14.644 |
| 9.25 | 14.61 |
| 10.25 | 14.4 |
| 11.153 | 14.119 |
| 12.221 | 13.628 |
| 12.772 | 12.907 |
| 13.145 | 12.141 |
| 13.495 | 11.229 |
| 13.345 | 11.695 |
| 13.827 | 10.661 |
| 14.051 | 10.365 |
| 14.508 | 9.962 |
| 15.022 | 9.491 |
| 15.719 | 9.117 |
| 16.004 | 8.912 |
| 16.532 | 8.611 |
| 17.056 | 8.132 |
| 17.493 | 7.762 |
| 17.821 | 7.502 |
| 18.183 | 7.157 |
| 18.479 | 6.835 |
| 18.859 | 6.611 |
| 19.108 | 6.258 |
| 19.451 | 6.091 |
| 19.8 | 5.876 |
| 20.068 | 5.639 |
| 20.118 | 5.5 |
| 20.276 | 5.268 |
| 20.542 | 4.932 |
| 20.592 | 4.736 |
| 20.642 | 4.678 |
| 20.676 | 4.5 |




Equivalent point volume: 20.676 ml

Equivalent point volume of blank: 21.778 ml

SAP =56.1 x.113 (21.778 - 20.676) /20.0193 = 0.35 mgKOH/ g