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Neutron Activation Analysis (NAA) of Quality of Tellus 68 Lubricating Oil for Effective Cooling of Turbo Generators

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Abstract: *This work involves the use of experimental procedures of neutron reflection process, irradiation and instrumental neutron activation analysis (INAA) for property characterization of Tellus 68 lubricating oil. The total hydrogen content of two samples of Tellus 68 lubricating oil was determined using 37 GBq^{241} Am-Be source. Further procedure requires the minimum sample preparation, two irradiation regimes and four counting strategies adopted on the basis of half-life of product radionuclide and the neutron spectrum parameters in the inner and outer irradiation channels of Nigeria Research Reactor (NIRR-1). Samples were prepared by pulverising, homogenising, mass determination and packaging. Pre- irradiation sample treatment (such as cleaning, drying or ashing, pre- concentration of elements of interest for elimination of interfering elements, sub- elements, sub- sampling and packaging) were carried out. Irradiation (prompt γ –ray counting in PGNAA) and radiochemical separation (only in RNAA) was conducted. Radioactive elemental concentration was measured for calculation of results for preparation of the NAA result. The analytical results of instrumental NAA of the oil samples when compared with manufacturer's specification validates Tellus 68 lubricating oil to be of good quality for effective cooling of Turbo generator.*

Keywords: *Detection Limits, Hydrogen Content, INAA, NAA irradiation; NIRR-1.*

1. INTRODUCTION

Turbo generators are designed to accommodate rise in temperature of the bearing oil and bearing pads when the generator is running for continuous power generation. Tellus 68 lubricating oil was investigated (Adaramola et al., 2017) to be suitable for removal of the heat generated on the upper guide bearing and to be kept at normal working temperature of 60 ^{0}C. The thrust of this work is to employ neutron reflection technique, short-life and long life irradiation for verification of the suitability of Tellus 68 lubricating oil for use in hydro power turbo generators. Lubricant application has been based on their chemical and physical properties for which successful techniques of neutron reflection, scattering and transmission methods used for bulk analysis of petroleum products (Csikai et al., 1999). The first stage of the design and experimental setup explores thermal neutron reflection applied for samples of common oils and improved neutron reflection method used by investigators (Akaho et al., 2001; Jonah et al., 1997) for the evaluation of hydrogen content of Tellus 68 lubricating oil.

Hiroshi et al. (1983) adopted the simultaneous utilization of neutrons and $\gamma - rays$ from ^{252}Cf for measurement of moisture and density of wax content in crude oil. Akaho *et* al, 2002 combined neutron transmission and reflection techniques for the classification of crude oil samples and Clare *et* al, 2001 employed molecular simulation for the property characterisation of the viscosity –temperature dependence of lubricants.

The first neutron reflection facility installed in the Centre for Energy Research and Training (CERT) Zaria "Nigerian Research Reactor-1 (NIRR-1)" was calibrated for multi-elemental analysis of over 30 elements to standardise routine INAA (Jonah et al., 2006). The two features of neutron induced reaction - high penetrability for neutrons and gamma radiation - ensure that its standardisation is potentially easy and accurate. As the signal to concentration ratio is nearly matrix independent, the sample preparation is rather easy; therefore, the risk of systematic or random errors is reduced. A relative standardisation can be performed by means of individual mono-element standards, or by using synthetic or natural multi-element standards. Following the NIRR-1

Standardization it was used in this work for high efficiency determination of a number of main-components and trace elements in the two samples of Tellus 68 lubricating oil.

With $^{3}He(d,n)^{4}He$ nuclear reaction fast were generated and the energy of the produced mono-energetic neutrons was 14 MeV thermal neutron (Yousif et al., 1995) for the investigation of neutron fields used in elemental analysis of bulk samples. The typical neutron yields of about 10^{11} neutrons s^{-1} mA^{-1} means a neutron flux of approximately 10^{9} neutrons cm^{-2} s^{-1}. The peak location on sample R is a measure of the gamma energy while the peak area is proportional to the photon emission rate. High energy pulse on scale shows the full-energy-peak efficiencies of the source- detector system.

2. METHODS

The Nigeria Research Reactor-1(NIRR-1) at the Centre for Energy Research and Training (CERT), ABU Zaria was used for NAA of Tellus 68 lubricating oil samples. The INAA procedure requires the minimum sample preparation, two irradiation regimes and four counting strategies adopted on the basis of half-life of product radionuclide and the neutron spectrum parameters in the inner and outer irradiation channels of NIRR-1. The adopted procedures eliminate the use of flux monitors and standards during irradiation of unknowns. This reduced the cost of NAA, increase sample throughput and improve the quality of analytical data.

2.1 Determination of Hydrogen Content in Tellus 68 Lubricating Oil

The Centre for Energy Research and Training (CERT), A.B.U, Zaria neutron reflection facility was used to determine the total H+ content in the two samples of the lubricating oil analyzed. The neutron source is embedded in the paraffin wax moderator and the sample container placed directly on the source. The indium are fastened to the bottom of the sample container and covered by 1 cm thick cadmium lining to cut off fast neutrons emanating from the moderator. This arrangement ensured that only the thermal neutrons from the sample reach the detector. The sample container was filled with 0.6 litre of oil sample and placed on the neutron source. The sample was exposed to fast neutrons for one hour to induce adequate activity to the indium foil. After the irradiation, a relaxation or cooling time of 20minutes was then allowed for the activities of nuclides with short half –life to decay.

The radioactivity measurement of the foil was carried out for the next 30 minutes (acquisition period). 500 cl of samples and hydrocarbon standards was taken in sample container and counted. Each sample and standard was counted 10 times for 10 seconds each, and the average neutron count, I was deduced. The neutron count without any sample was taken 10 times for 10 seconds and the average count without any sample, I_0 was deduced in the experimental setup shown in Figure 1. Finally the physical density, ρ of samples R (Regenerated Tellus 68 lubricating oil) and F (Fresh Tellus 68 lubricating oil) were measured and recorded in Table 1.

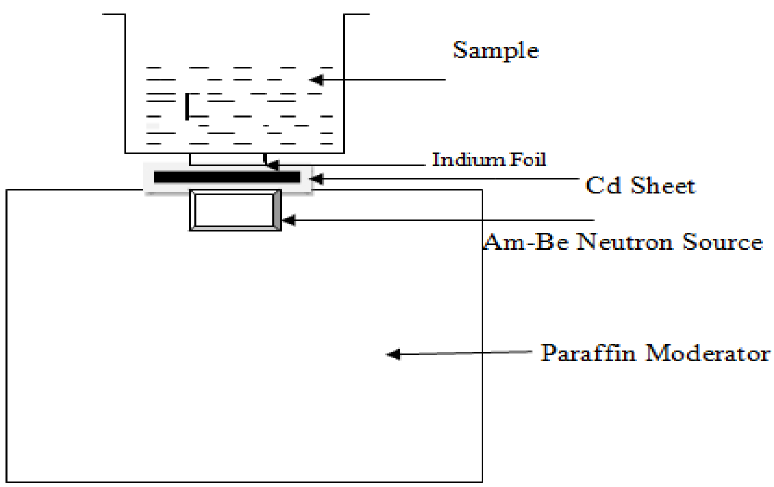

Figure 1: Experimental set-up for Determination of Hydrogen Content of Tellus 68 lubricating oil samples

2.2 NAA Short- Life Irradiation of Samples

Two samples of Tellus 68 lubricating oil were packed and sealed in 7 cm^3 rabbit capsules and sent for irradiation in turn in an outer irradiation channel B4, where the neutron spectrum is soft. The outer irradiation channel was chosen to eliminate corrections due to nuclear interferences caused by threshold reactions, notably Magnesium (Mg) in the present of Gold (Au); Au in the present of Chlorine (Cl), and Sodium (Na) in the presence of Phosphorous (P). This is due to the proximity of the inner channels of MNS reactors core leading to relatively higher ratio of fast thermal neutrons.

For the short-lived irradiation regime, the 1st round of counting was performed for 10min (i.e. S1) after a waiting time of 2-15 minutes. Samples were placed on a plexi-glass sample holder designated 'H2' which corresponds to source- detector geometry of 5 cm. The 2nd round of count was also carried out for 10 minutes, following the short irradiation regime (i.e. S2) after a waiting period of 3-4 hrs. Samples were counted on a plexi-glass sample holder designated 'H1' corresponding to source- detector geometry of 1cm. The neutron flux setting for the short-lived irradiation was raised to 5×10^{11} n/cm^{2}s in order to increase the detection sensitivities for analysis of elements using procedures S1 and S2.

2.3 NAA Long - Life Irradiation of Samples

The samples for long-life activation were wrapped in polythene bags films and packed in a stack inside 7 cm^3 rabbit capsules and sealed for irradiation. Samples were irradiated for 6 hrs in any of the smaller inner irradiation channels (i.e. A1, B1, B2 and B3) to take advantage of the maximum value of thermal neutron flux in the inner channels. The neutron flux variability over irradiation volume was determined experimental to be less than 2% through measured specific activities of irradiated Cu wires arranged axially and radially inside the vial. The stability of neutron flux for a long period of irradiation was checked by monitoring the neutron flux reading of a fission chamber connected to the micro-computer control system. Results indicate a stable neutron flux over irradiation period. Radioactivity measurement of induced radionuclides was performed by the PC-based gamma ray spectrometry set-up.

3. RESULTS AND DISCUSSION

3.1 Determination of Hydrogen Content in Lubricating Oil

The result of neutron reflection technique was used to determine the total H+ content in the samples of the lubricating oil used and this is presented in Table 1.

Table 1: Neutron Reflection Parameter and Hydrogen Contents

Reagents	Average neutron count, I	Density ρ (kg/m^3)	Hydrogen (wt. %) H	Reflection Coefficient, η
$C_6H_4(CH_3)_2$ xylene	1221	0.864	9.43	0.99685
CH_2 (Paraffin oil)	1263	0.880	14.29	1.3204
n-brt alcohol	1295	0.789	13.514	1.23458
C_6H_6 (Benzene)	1299	0.879	7.69	1.11511
C_2H_5OH (Ethanol)	1314	0.79	13.04	1.26968
Methanol	1274	0.792	11.77	1.18948
C_6H_{14} (n-hexane)	1336	0.66	16.28	1.57058
H_2O	1362	1.0	11.11	1.0762
Sample R (Regenerated Oil)	1394	0.86323	13.5656	1.3032
Sample F (Fresh Purified Oil)	1342	0.90686	10.9322	1.15313
I_0	656			

The relation between the neutron reflection parameter denoted ***η*** and the count rate with and without the sample is given by the formula,

$$\eta = \frac{I - I_0}{I_0} \quad \text{(Akaho et al., 2001)} \quad (1)$$

Where I is the average neutron count with samples and standard, while

I_0 is the average neutron count without samples and standards.

Figure 2 show the graph of neutron reflection parameter plotted against the sample containing known H (wt. %) using the following expression:

$$\eta = \eta_o + m\,H \text{ (wt. \%)} \quad (2)$$

where η_o= 1.96957 and m = 0.23351

The hydrogen content presented in table 1 is the average values of the data obtained using the peak analysis reports of 1097.3 and 1293.5 keV gamma lines respectively. Data in Table 1 were used to construct the calibration line of neutron reflection parameter against the hydrogen content in oil samples. The solid line for determination of total hydrogen content was derived by fitting Equation 2**,**
$\eta = \eta_o + m\,H$ (wt. %)

where ηo= 1.96957 represents the effect of the matrix and *m* = 0.23351 is the slope of the calibration line for hydrogen content as shown in figure 2.

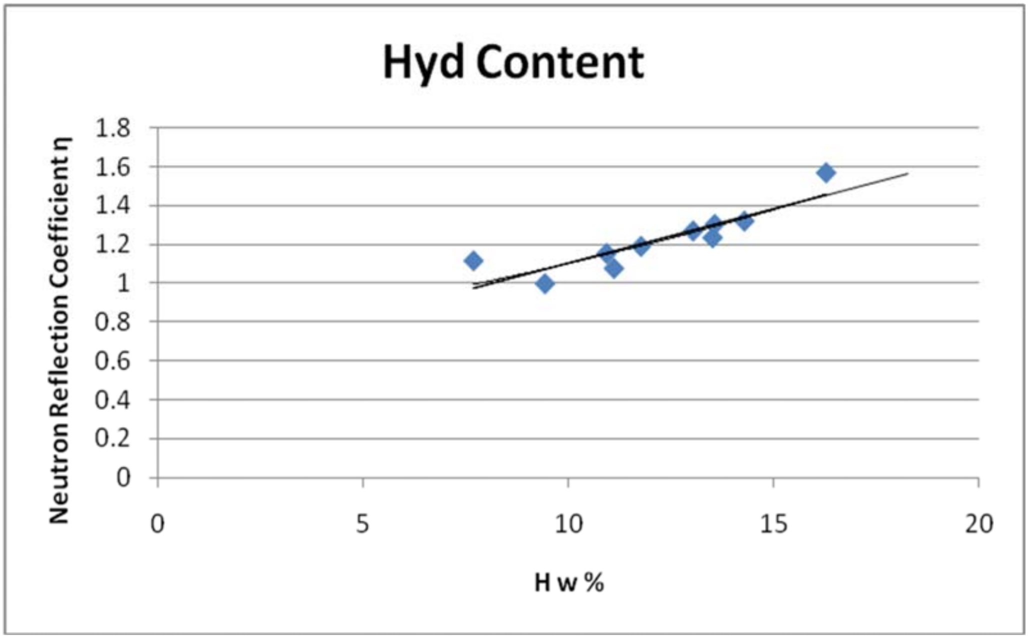

Figure 2: Neutron reflection parameter -Hydrogen Content Graph

3.2 Summary of NAA Data Analysis

The analytical results of instrumental NAA of two (2) oil samples presented in Table 2 and Table 3 is based on thermal neutron reaction from NIRR-1 in combination with high resolution gamma-ray spectrometry. This NAA method allows a wide range of elements to be measured in the samples with high accuracy and precision with no contamination and no elaborate sample preparation.

Table 3: Element Concentration in Oil Samples

Element	Sample F	Sample R	Element	Sample F	Sample R
Mg (ppm)	NA	NA	**Fe (ppm)**	BDL	BDL
Al (ppm)	14.7 ± 1.3	13.8 ± 0.3	**Co (ppm)**	BDL	BDL
Ca (ppm)	40650 ± 9024	35280 ± 8961	**Zn (ppm)**	208 ± 5	383 ± 7
V (ppm)	BDL	0.16 ± 0.03	**Br (ppm)**	BDL	0.08 ± 0.02
Mn (ppm)	0.18 ± 0.03	0.23 ± 0.02	**Rb (ppm)**	BDL	BDL
Dy (ppm)	BDL	BDL	**Sb (ppm)**	BDL	BDL
Na (ppm)	2.5 ± 0.2	4.6± 0.02	**Cs (ppm)**	BDL	BDL
K (ppm)	BDL	BDL	**Ba (ppm)**	BDL	BDL
As (ppm)	BDL	BDL	**Eu (ppm)**	BDL	BDL
La (ppm)	BDL	0.023± 0.006	**Yb (ppm)**	BDL	BDL
Sm (ppm)	BDL	BDL	**Lu (ppm)**	BDL	BDL
U (ppm)	BDL	BDL	**Hf (ppm)**	BDL	BDL
Sc (ppm)	BDL	BDL	**Te (ppm)**	BDL	0.013
Cr (ppm)	BDL	BDL	**Th (ppm)**	BDL	BDL

BDL: - Below Detection Limit NA: - Not Analyzed

The reported uncertainty (NA- Not Analysed and BDL-Below Detection Limit) was mainly from counting statistics and is not the normal standard deviation on respective analysis on replicate analysis.

Table 4: Manufacturer's Oil Specification

S/No	Property	Specified Value
1	Viscosity at 50 °C	30-37 cSt
2	Flash Point	min -160 °C
3	Pour Point	min – 20 °C
4	Neutralization Number	0.3 mg KOH/g
5	Water content by volume	0.1%
6	Ash Content	0.02

4. CONCLUSIONS

The hydrogen content is a reflection of the neutralization number tendency to form acid. For TELUS oil sample R (Regenerated oil), the H wt. 13.566% which is an indication of acidity implies neutralization number is 0.136 while for the purified lube oil sample F (Fresh purified lubricant) the H+ wt. is 10. 932% indicating a lower neutralization number of 0.109 a better and lower tendency of acidity. The hydrogen content of regenerated lubricant increases with usage as shown in the results indicating the possibility of using this set up as quality control facility. Also higher H+ content reduces the flash point and the flammability of lube oil as increased H+ content cause more water content which eventually result in lower oil viscosity.

Al, Ca, Mn and Na were present in higher concentration in fresh oil than in used regenerated oil as expected. The presence of Na, Ca and Mn ions in lubricating oil offers protection from metal rust and acts as emulsifiers making mineral oil miscible with water. Zn content in fresh oil is however lower concentration indicating lower ash content impurities than in regenerated oil. Va, La and Br presence also shows impurities such as ash. Al and Cl presence are extreme pressure additive preventing seizure and welding between metal surfaces under conditions of extreme pressures and temperatures increasing emulsion life of oil. Their presence also prevent odour during operation of machinery.

The discrepancies between the measured and the manufacturer's specified values for regenerated oil in this work can be attributed to the presence of impurities in form of trace element such as Se, As and Sb which are reflection of ash content; while O and H are indication of moisture content. Table 3 and Table 4 clearly show Te which increase the acidity of lubricating oil was not detected (ND) hence the oil is suitable for use for cooling the turbo generator.

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