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Radioactive Dating of an Artifact: A Comparative Approach

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ABSTRACT

The methods through which ages of artifacts can be unraveled have been highlighted - most notably carbon-14 dating. Carbon-14 dating is a way of measuring the age of certain archeological artifacts of biological origin up to $5x10^5$ years old (BBC, 2005). By employing this dating method, the researchers determined the age of a semi-prehistoric bone (antler). The Liquid gas Scintillation Counter (LSC) - Ansitron-1300 was used to monitor the activity of beta-particles in the bone. The mean beta-particle count was then obtained to be 14.75cpm. From the experimental results obtained and further comparative calculations using a modified *Activity-Time equation*, $A(t)=15\exp\{(-1.2097x10^{-4})t\}$ and *Libby's equation*; $t = -T_{1/2}\ln\{1+ (\Delta/1000)\}/0.693$; the dates of (138.96 ± 0.48) and (139.24 ± xxx) years respectively were obtained for the bone (antler). Thus, the results agree within the ambit of experimental research. Hence the approach provides an effective method for dating radioactive artifacts of biological origin. The research therefore finds useful applications in judicial verdicts to unlock land related disputes in which human artifacts could be recovered from involved sites. Moreover, the equivalent absorbed dose rate, H' of the laboratory was also estimated to be 2.84 rems/week. This is however, higher than the 0.25 rems/week Maximum Permissible Dose (MPD) recommended for workers in radiation laboratories by the International Commission on Radiological Protection (ICRP, 2007). Adequate shield is thus; emphasized for the personnel working in this laboratory.

Keywords: *Carbon-14, Radioactive Dating, Artifact*

1. INTRODUCTION

The question "How old are you?" is often one of the first questions that comes the mind of historians when meeting someone. The same is true in science and other fields when encountering a new sample or unknown specimen. However, a fossil, wood, rock, thunderbolts or elf-shot will not answer the above question and so many useful analytical methods have been devised out of man's curiosity to place an age on these objects. For instance, by the Middle Ages "Magic pots" (probably cremations urns) that mysteriously emerged from the ground during erosion or by the action of burrowing animals intrigued the people of India and Europe. In a related development, the discovery of sculptures in Greece and Rome raised more probe. Thus in the 16th century, some scholars in North-Western Europe had began to realize that information about the ancient past could be derived from the study of the field monuments - this marked the beginning of dating (Encarta, 2004).

Appraisal and Theory of Carbon-14 Dating

Dating is a process involved in finding or obtaining the age of past events (Bradley, 1985). In trying to uncover the dates of artifacts, scientists have over time employed various techniques-some historical and others scientific. Historical dating is based on the equation of the archaeological records with dates provided in written chronologies and calendars compiled by ancient people. Historical technique of dating is grouped into relative and absolute methods (Britannica, 2010).

Scientific dating on the other hand employs the measurement of time-dependent parameters to obtain the age of artifacts. It can be sub-classified into radiometric, chemical, biological, magnetic, varve analysis and astronomical dating (Higham, 2005).

Carbon-14 dating therefore is a radiometric dating technique that uses the naturally occurring isotope of carbon to determine the age of carbonaceous material of biological origin. It was discovered by William F. Libby and his colleagues in 1949 (Mc Graw-Hill Encyclopaedia, 2004). According to Libby as quoted in Bradley (1985), the radio-carbon age (RCA) of a geological or archaeological specimen is given by:

$$T = -\frac{T_{\frac{1}{2}}}{0.693}\ln\left[1+\left(\frac{\Delta}{1000}\right)\right] \qquad (1)$$

where $T_{1/2}$ is the half life of radiocarbon (5730 ± 40years), Δ is a correction factor define as:

$$\Delta = \left(\delta^{14}C\right)_{\max} - 2(\delta^{13}C + 25)\left(1+\frac{\left(\delta^{14}C\right)_{\min}}{1000}\right) \qquad (2)$$

where $\delta^{13}C$ and $\delta^{14}C$ are the respective fractionation effects in ^{14}C and ^{13}C during sample decay.

$$\delta^{14}C = \left(\frac{A_{Sample} - 0.95A_{Ox}}{0.95A_{Ox}} \right) * 1000$$

(3)

where A_{sample} is the ^{14}C activity of the sample corrected from background radiation and A_{Ox} is the 1950 activity of the National Bureau of Standard Oxalic Acid corrected from background radiation and isotopic fractionation.

To curb the difficulty in the calculation of the fractionation effect ($\delta^{14}C$) due to the activity value of oxalic acid standard (A_{Ox}), Stuiver and Polach (1977) offered an approximate formula which agrees with equation (3) and the plot in fig 1.

$\delta^{14}C$ = percentage change in % A_{Sample} * 1000

(4)

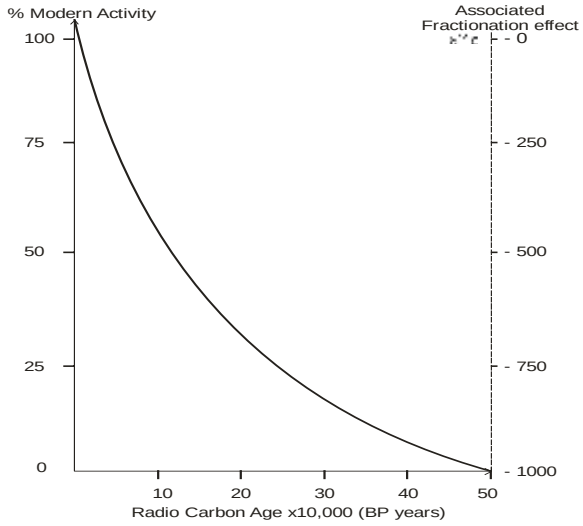

Fig1. Plot of $\delta^{14}C$ Estimate against Percentage Activity and Radio Carbon Age of an Artifact (Stuiver & Polach, 1977)

Using these equations, time related problems have been deciphered in anthropology, oceanography, pedology and climatology (Stuiver & Polach, 1977). However in this research, the comparative approach of radio-carbon dating is employed using a modified *Activity-Time* equation and the *Libby's equation*.

Theory of carbon-14 Dating

Carbon as a compound has three basic isotopes ^{12}C, ^{13}C and ^{14}C (radioactive isotope). The most common of these is C-12 with predominant percentage of 99% of all the carbon atoms in plants and animals. C-13 comprises of about 1% of the total. The third C-14 is found in tiny amounts compared to others. In a living thing, there is only one C-14 atom for over trillion C-12 atoms (Gore, 1999). The radioactive carbon (C-14) is produced when cosmic ray protons blasts nuclei in the upper atmosphere producing neutrons which in turn bombard nitrogen, the major component of the atmosphere (Nave, 2003).

$$^{14}_{7}\mathrm{N} + {}^{1}_{0}\mathrm{n} \rightarrow {}^{14}_{6}\mathrm{C} + {}^{1}_{1}\mathrm{H}$$

(5)

The radioactive carbon (C-14) is then acquired by plants from carbon (IV) oxide (CO_2) in the atmosphere during the process of photosynthesis. Animals thus get the carbon as a result of heterotrophic feeding on plants and respiration. Hence all plants and animals (living) have a constant ratio of C-14 to C-12 which stands at:

C-14/C-12 = 1.3×10^{-12}

(6)

This ratio according to Gore (1999) equals that in the atmosphere. However when the organism dies, the intake of carbon is stopped. The radioactive carbon (C-14) atoms therefore decays at a constant rate of (5730 ± 40) years (porter, 2004) emitting beta-particles and thus converted to Nitrogen-14 (Pitman et al, 2004).

$$^{14}_{6}C \rightarrow {}^{14}_{7}N + {}^{0}_{-1}e$$

(7)

Therefore, the number of carbon-14 atoms counted per minute in an artifact gives a function of the time elapse (Brain, 2004).

$$N(t) = N_o e^{-\lambda t} \quad (8)$$

where N_o is the initial number of C-14 atoms in the sample, N(t) is the number of residual C-14 atoms from the sample at an instant of time, λ is a constant dependent on the half life of C-14 atom and t is the time elapsed since the death of the sample organism.

Since the size or weight of an atom does not indicate how much radioactivity it contains (REAC, 2002), the activity of a radio isotope which is the number of disintegrations per second is the considerable quantity. The activity is determined with the aid of radiation detectors such as Geiger-Muller tube, spectrometer, Scintillation counters, dosimeters, etc.

According to experiments on radioactive sources, the activity (dN/dt) at an instant of time, *t* of radioactive substances is proportional to the number of atoms *N*(t) present in the sample (Young et al, 1996).

$$\frac{d}{dt}N(t) \alpha N(t) \text{ or } \frac{d}{dt}N(t) = -\lambda N(t) \quad (9)$$

where -λ is the decay constant.

Solving the differential equation (9) by separation of variables leads us to equation (8) and substituting A(t) = -N(t)λ we have

$$A(t) = A_0 e^{-\lambda t} \quad (10)$$

where A_o is the initial activity of C-14 in the sample at *t=0*, A(t) is the residual activity of C-14 from the sample at any instant of time *t*, λ is a constant dependent on the half life of C-14 atom and *t* is the time elapsed since the death of the sample organism.

According to Nave (2003), A_o = 15.0 counts per minute. And at t = $T_{1/2}$ (half life), $A(t) = \frac{A_0}{2}$ which leads equation (10) to equation (11):

$$\lambda = 0.693/T_{\frac{1}{2}} \quad (11)$$

Substituting equation (11), $T_{1/2}$=5730 years and A_0 = 15.0 cpm into (10) yields the modified *activity-time* equation (12).

$$A(t) = 15e^{-(\frac{0.693}{5730})t} \approx A(t) = 15e^{-1.2097x10^{-4}t} \quad (12)$$

Equation (12) explicitly expresses the activity in an artifact as a function of time. Therefore, the age of an artifact could be found if the beta particles' activity of radiocarbon in it at an instant is known.

Detection of Beta-Particle Activity

The theory of beta decay is based on the concepts of neutron or proton conversion; a nuclear process which converts radioactive isotope to a more stable state. This transmutation is given as (Murray, 1993):

$$n \rightarrow P^+ + e^- + v, \qquad P \rightarrow n + e^+ + \tilde{\nu}\bar{\nu} \quad (13)$$

where P is proton, n is neutron, e^- is electron, e^+ is positron, v is neutrino and $\tilde{\nu}\bar{\nu}$ is the antineutrino.

In beta radiation, the maximum energy is the algebraic sum of the energy of the two emitted particles (i.e the beta particle and the antineutrino) (Britannica, 2010).

$$E_{max} = E_{beta\ particle} + E_{antineutrino} \quad (14)$$

Hence only few of the emitted beta particles have appreciable proportion of energy; as the energy is shared between the two particles, with beta particles having approximately only one-third of the maximum energy (Britannica, 2010).

$$E_{beta\ particles} = {}^1/_3\ E_{max} \approx 0.156MeV \text{ (for C-14)} \quad (15)$$

This low energy content of the beta particle poised great threat to its detection and measurement by radiation detectors until the invention of the liquid scintillation counter by Noakes in 1965 (Aitken, 1990).

Materials

Liquid gas Scintillation counter (Beta detector), Agate mortar and pestle, spatula, beakers, 60% Hexaoxochlorate (VII) ($HClO_4$), Hydrogen Peroxide (H_2O_2), Ethoxyethane ($C_4H_{10}O$), benzene (C_6H_6), conical and flat bottomed-flasks, forceps, Sodium Hydroxide (NaOH) base, Hydrochloric (HCl) Acid, rubber tubing, Standard Oxalic Acid (optional) and the antler (i.e the artifact obtained from ancient semi-prehistoric site).

2. METHODOLOGY

Sample Collection

The artifact (Antler) used in this research work was obtained from ancient semi-prehistoric family gallery from Ushongo Local Government area of Benue State; Nigeria. According to the oral history from the head of the gallery, the artifacts have been kept for about 200years. Hence these could serve as suitable specimens for the radioactive dating research. The gallery according to him was a memorial established by his ancestral fathers who were predominant game hunters as well as herbal doctors. The artifacts therefore served as items of fame, traditional medicine practices and markers of dates associated with records of events, births or bereavement of family relations. Among the artifacts were sculptured smoking pipes, wooden chairs and beds, wooden dishes, clay pots, beads, hides and skin, furs, calabashes, charcoal, roots, feathers, skinned bags, shells, drums, bones of domestic and hunted animals from which the deer's antler was gotten for the experiment.

Sample Preparation

Using the Hydrochloric Acid (HCl), Sodium Hydroxide (NaOH) and Hydrogen Peroxide (H_2O_2) solutions; the bone was pretreated to extract the impurities present thus eliminating the problem of sample contamination. The pretreated bone was then allowed to dry at 30°C, after which it was pounded using the agate mortar/pestle. The sample was divided into two portions of 140g each (Test and confirmed experiments) then suspended in a solution of 0.6M (60%) Hexaoxochlorate (VII) ($HClO_4$), 0.05M Ethoxyethane ($C_4H_{10}O$), 2.0M benzene (C_6H_6) and allowed to stand undisturbed for 10 minutes.

Calibration of Equipment

The liquid gas scintillation counter with model name Ansitron - 1300 used in the detection of the radiation from the artifact (bone) has been calibrated at the factory to an accuracy of 0.01 count per minute and delivers radiation pulses of 5mV or above. However, it is subject to recalibration/readjustment through the knobs and the timer circuits to give readings in counts per second. During the experiment, readings are usually taken on the graduated scales within the specific time intervals. The device utilizes voltage of 800-1500v to operate depending on the type of radiation one wants to detect.

Measurement of Beta Radiation

In using the scintillation counter, the readings were taken in counts per minutes. The Scintillation machine was booted and allowed to initialize its settings. Without placing any sample in the chamber of the machine, the background radiation of the laboratory was read from the counter and recorded after which the test sample was placed in its combustion chamber then beta emission/count initiated. The measurement was repeated on the confirmed experiment and results obtained. The readings of the beta counts were taken in intervals of 1 minute on the graduated scales without interrupting the set up in order to minimize errors due to fractionation effect. However, in taken the experimental readings in table 1, some counts whose deviations were too large were rejected. The actual beta radiation and activity from the bone were then obtained after correction; by deducting the background radiation from the measured values presented in table1. This result is therefore presented in table 2.

3. TEST RESULTS

Table 1: Experimental Readings for the Emitted Beta Count (uncorrected)

Mean background radiation of the laboratory $\bar{\delta} = 2.80$

Time (min)	Beta Counts (β)	Count rate A(t) = β/t (cpm)
1.0	17.50	17.50
2.0	32.40	16.20
3.0	47.02	15.67
4.0	61.84	15.46
5.0	76.05	15.21
6.0	91.30	15.21
7.0	105.77	15.11
8.0	121.20	15.15
9.0	135.73	15.08
10.0	149.20	14.92
11.0	165.38	15.03
12.0	179.80	14.98
13.0	194.42	14.95
14.0	208.60	14.90
15.0	223.15	14.87
16.0	239.60	14.97
17.0	254.06	14.94
18.0	268.50	14.91
19.0	283.62	14.92
20.5	298.00	14.90
21.0	313.60	14.93
22.0	327.30	14.87
33.0	342.28	14.88
24.0	335.60	14.81
25.0	375.30	15.01

Table 2: Experimental Readings for the Emitted Beta Count (corrected)

Mean background radiation of the laboratory $\bar{\delta} = 2.80$

Time (min)	Beta Counts (β-δ)	Count rate A(t) = (β-δ)/t (cpm)
1.0	14.70	14.70
2.0	29.60	14.80
3.0	44.22	14.74
4.0	59.04	14.76
5.0	73.25	14.65
6.0	88.50	14.75
7.0	102.97	14.71
8.0	118.40	14.80
9.0	132.93	14.77
10.0	146.40	14.64
11.0	162.58	14.78
12.0	177.00	14.75
13.0	191.62	14.74
14.0	205.80	14.64
15.0	220.35	14.69
16.0	236.80	14.80
17.0	251.26	14.78
18.0	265.70	14.75
19.0	280.82	14.78
20.5	295.20	14.76
21.0	310.80	14.80
22.0	324.50	14.76
33.0	339.48	14.76
24.0	332.80	14.70
25.0	372.50	14.90

NB: some readings were rejected due to their large degree of deviation.

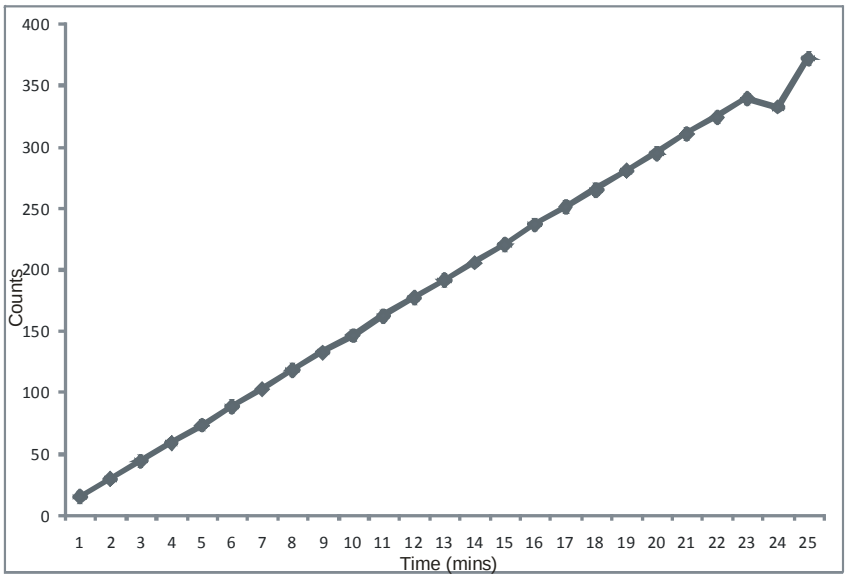

Fig 2.Count versus Time Plot for the Corrected Beta Radiation

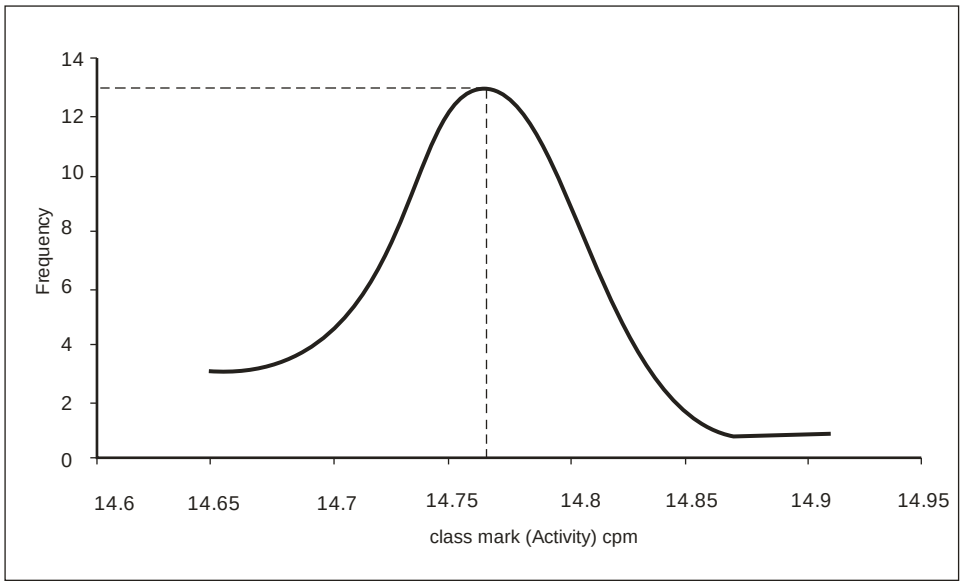

Fig 3. Frequency – Activity Plot for the Corrected Beta Counts

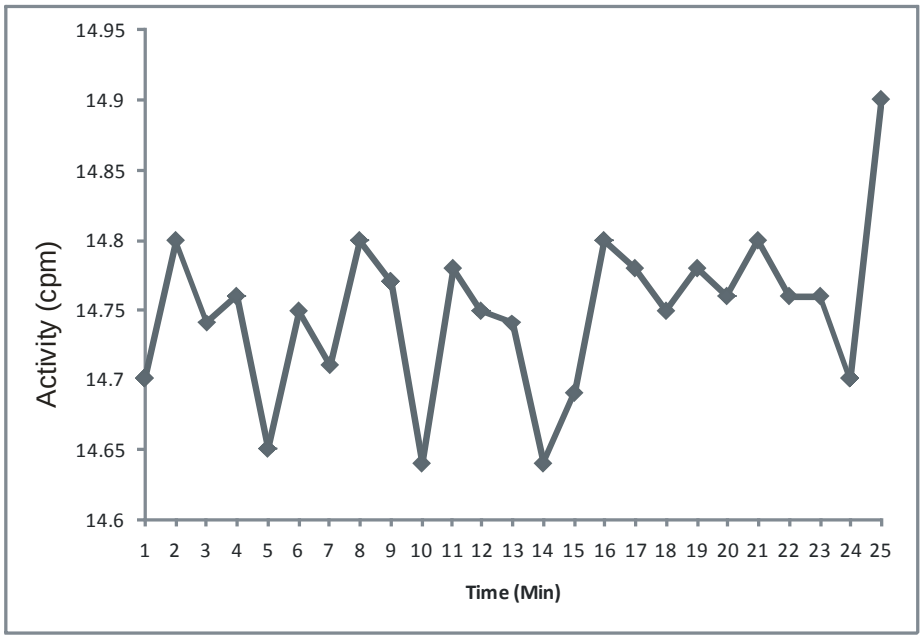

Fig 4. Activity – Time Plot for the Corrected Beta Counts

4. ANALYSIS OF RESULTS

In this research work, the statistical analysis is adopted since radioactive decay is a discrete random phenomenon (Nelkon et al, 1995) which conforms to statistical parameters like mean, percentiles, standard deviation and variance.

Table 3. Frequency Distribution Table for the Corrected Radiation

Assumed Mean $\bar{A}$' = 14.81, Class Size, c = 0.05

Class Interval, A	Class Mark A'	frequency, f	fA	u = (A'-$\bar{A}$')/c	u^2	fu	fu^2

14.64 – 14.68	14.66	3	43.98	-3	9	-9	37
14.69 – 14.73	14.71	4	58.84	-2	4	-8	16
14.74 – 14.78	14.76	13	191.88	-1	1	-13	13
14.79 – 14.83	14.81	4	59.24	0	0	0	0
14.84 – 14.88	14.86	0	0	1	1	0	0
14.89 – 14.93	14.91	1	14.91	2	4	2	4
		$\sum f=25$	$\sum fA=368.85$			$\sum fu= -28$	$\sum fu^2=60$

Mean Distribution of the Beta Particles (Activity), $\bar{A} = \frac{\sum fA}{\sum f} = \frac{368.85}{25}$ = 14.75cpm (98.33%)

The Standard Deviation of the particle distribution $\sigma = c\left[\frac{\sum fu^2}{\sum f} - \left(\frac{\sum fu}{\sum f}\right)^2\right]^{\frac{1}{2}} = 0.05\left[\frac{60}{25} - \left(\frac{-28}{25}\right)^2\right]^{\frac{1}{2}}$

= 0.054 cpm

The Variance of the particle distribution, $S = \sigma^2 = (0.535)^2$ = 0.00286

The standard error in the activity level therefore, $S_e = \frac{\sigma}{\sum f} = \frac{0.054}{25}$ = 0.00216 cpm (0.0142%)

Therefore the activity of the artifact under observation is:

A(t) = (14.75±0.00216) cpm = (98.33±0.0142)% cpm

I. Age Calculation

(a) From the modified activity equation (12), the age of the artifact is calculated thus:

$$A(t) = 15e^{-1.2097x10^{-4}t}$$

Solving for t, we have,

$$t = \frac{10^4}{1.2097}\ln\left[\frac{15}{A(t)}\right] = \frac{10^4}{1.2097}\ln\left[\frac{15.00}{14.75}\right] \approx 138.96 \text{ years}$$

(b) Using Libby's equation (1) and the Stuiver-Polach approximation (4) with fig1,

$$T = -\frac{T_{\frac{1}{2}}}{0.693}\ln\left[1+\left(\frac{\Delta}{1000}\right)\right]$$

$$\Delta = \left(\delta^{14}C\right)_{max} - 2(\delta^{13}C + 25)\left(1 + \frac{\left(\delta^{14}C\right)_{min}}{1000}\right)$$

For maximum age of an artifact, $(\delta^{14}C)_{min}$ = - 1000 (see fig1) and $\Delta = (\delta^{14}C)_{max}$ - 0. Therefore,

$\Delta = (\delta^{14}C)_{max}$ = percentage change in %A(t)*1000 = (98.33-100)% x 1000 = -16.70

Therefore, with $T_{1/2}$ = 5730 years,

$$T = -\frac{5730}{0.693}\ln\left[1+\left(\frac{-16.70}{1000}\right)\right] = 139.24 \, years$$

II. Error

From the mean activity obtained, we have the associated standard error of ±0.00216 cpm. Therefore, to obtain the associated error in the age of the artifact, we use:

$$t^+ = \frac{T^+_{\frac{1}{2}}}{0.693}\ln\left[\frac{15}{A^+(t)}\right] \text{ and } t^- = \frac{T^-_{\frac{1}{2}}}{0.693}\ln\left[\frac{15}{A^-(t)}\right] \quad ***$$

Where $T^+_{1/2}$ = 5730 + 40 = 5770 years (half-life of carbon)

$T^-_{1/2}$ = 5730 - 40 = 5690 years

$A^+(t)$ = 14.75 + 0.00216 = 14.75216 cpm

$A^-(t)$ = 14.75 – 0.00216 = 14.74784 cpm

Substituting the values in ***, yields: t^+ = 138.72 years, t^- = 139.20 years

Hence the error in age $\delta t = \Delta(t^-, t^+)$ = 139.20 – 138.72 = ± 0.48 years

Therefore the age is = t ± δt = (138.96 ± 0.48) years

III. Estimation of Dose Equivalent, H

The Dose Equivalent, H (rems) is defined as:

$H = DQ_F$

where

Q_F is the quality factor = 1.7 for β-particles (Murray, 1993) and D is reduction absorbed Dose (Rad) defined by:

D = C x 6.0 x10-5; C is the count rate in counts per seconds (cps).

Hence from the table, the mean count rate of the Background radiation, C = 2.80/60 = 0.046 cps

Therefore, H = 0.046 x 6.0 x 10^{-5} x 1.7 = 4.69 x 10^{-6}rems.

The equivalent weekly absorption dose rate H' by a worker in such a laboratory is estimated thus:

Equivalent dose rate H' = 4.69 x 10^{-6} x 3600 x 24 x 7 = 2.84 rems/week.

IV. DISCUSSION OF RESULTS

Tables 1 and 2 present the experimental readings for the uncorrected and corrected beta counts obtained from the artifact (antler). The background radiation accounted for the high count values shown in table1 as against the corrected values in table1.

Figure 2 is the plot of *Cumulative Beta Counts against Time* for the corrected radiation. It shows that the count, though not uniformly radiated; was continuous with time. The experiment was stopped when the deviations in the counts became obvious as seen on the distorted region of the curve. This is attributed to depreciation of beta particles in the sample.

Figure 3 is the *frequency distribution versus count rate* plot for the corrected beta radiation. The threshold point on the ogive shows the mean, median and modal count – rate in the experiment. It reveals the interval of time when maximum emission of beta particles was received by the scintillation counter as supported by previous works on beta particle's energy spectrum of carbon-14 isotope.

Finally, figure 4 is the *activity rate* of the beta particles as monitored by the scintillation counter. The plot shows that the beta activity is stochastic despite the fact that the count was continuous with time as shown in figure 2.

V. CONCLUSION

Within the limits of the experiment errors, the mean instantaneous beta activity, A(t) for the artifact and the laboratory background were respectively (14.75± 0.0216) cpm and 2.8 cpm at the time these measurements were taken. Using the activity value of the artifact, its age was computed comparatively from the modified *Activity–Time* equation; $A(t) = 15e^{-1.2097x10^{-4}t}$ and the *Libby's equation*; $t = -\frac{T_{\frac{1}{2}}}{0.693}\ln\left[1+\left(\frac{\Delta}{1000}\right)\right]$ to be (138.96 ± 0.48) and (139.24 ± xx) years respectively. These values agree considerably within the purview of experimental research. Thus, the method of radioactive dating used in this work stands of immense reliability in dating relatively old archaeological artifacts of biological origin. The research procedure can therefore be applied in judicial verdicts to unlock land related disputes in which human artifacts could be recovered from involved sites.

Moreover, the absorbed equivalent dose rate H' due to the background radiation of the laboratory was estimated to be 2.84rem/week. This value is however higher than the 0.25rem/week Maximum Permissible Dose (MPD) recommended by the International Commission on Radiological Protection (ICRP, 1957). Hence adequate shield is recommended for the personnel working in this laboratory.

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