AGE DETERMINATION OF LIMESTONE ROCKS LEANG - LEANG CAVE COMPILER THROUGH ACTIVITY MEASUREMENTS $^{14}$C USING LSC

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ABSTRACT

Age Determination of Limestone of Leang - Leang Cave Through Activity Measurements of $^{14}$C Using LSC. This research has been carried out by using limestone samples taken from Leang-Leang Cave, Maros. Limestone is one of the environmental samples with a constituent of calcite mineral ($\text{CaCO}_3$) which is derived from the remains of flora and fauna that has been weathered and petrified. Sample preparation was done physically and chemically. Preparation of chemically by using a mixture of NaOH with 30% H$_2$O$_2$ followed by a mixture of HClO$_4$ with 30% H$_2$O$_2$, and the last with HCl solution to produce a clean sample with a weight reduction of 3.36% - 4.44%. Carbonate matrix samples as CO$_2$ is produced by reaction with 85% H$_3$PO$_4$ and absorbed by 1M KOH solution as K$_2$CO$_3$. The total carbon in the sample solution is 0.6144 - 0.9696 grams obtained through titration method. Radiocarbon dating method based on the measurement of the specific activity of the samples obtained from the results of counts LSC (Liquid Scintillation Counter) Hidex 300 SL. The specific activity of both of samples were 1,609±0.0359 DPM/g C and 7,718±0.109 DPM/g C. Age of both of limestone samples which were calculated from the specific activity were 25,607,403 ± 919,305 years and 8457,792 ± 921,899 years for BG I and BG II, respectively.

Keywords: Limestone, LSC (Liquid Scintillation Counting), radiocarbon dating, specific activity.

INTRODUCTION

Indonesia is an archipelago located in a very strategic position in the equatorial region that connects the continents of Australia and Asia and the Pacific and Indian oceans. The climate is pleasant, varied natural wealth that make Indonesia as a country which has great potential in natural resources (SDA) to develop into a powerful country in the Asia - Pacific (Suhadi et al, 1998). One of the natural wealth of Indonesia located in South Sulawesi, especially those in areas such as Maros - Pangkep the karst rock stood firm. Maros karst region - Pangkep upper part of some major rivers, including the River upstream Pangkep Putre River and River Bantimurung/Maros (Prawitosari, 2011). According to Ahmad (2001) in Daryanto and Oktariadi (2009) Maros karst region is
part of a mountain range that stretches from Maros area to the north to the entrance to the area Pangkep. This area formed from the dissolution of limestone outside as the hills towering upright, and which occurs in the phenomenon which forms a very beautiful and unique, such as the formation of caves horizontal and vertical caves, stalactite, stalagmite, and others. Growing limestone is characterized by a more solid and resistant to denudation processes and form steep hills and higher elevations outside the karst formations in the form of a hill.

Organic limestone is a collection of residual flora and fauna that have died (fossil) and sedimented. In his lifetime flora and fauna require elements Ca, Mg, O and C are present in the water. Chemical processes that occur on the flora and fauna after death into a fossil and mineral crystals forming system without changing the physical form of fossils. In mineralogy of the fossil is composed of the mineral calcite (CaCO₃) and dolomite [CaMg(CO₃)₂]. In general, the fossil fauna containing calcite magnesium grading (4% -16% )mol MgCO₃, while the fossil flora of approximately (7,7% -28.75%)mol MgCO₃. Mineral calcite or dolomite mostly small (± 0,2 microns or more) is called carbonate mud (Harjanto, 2001). Because of the small size, the optical and physical properties relaitf same, so it is difficult to distinguish the two minerals. Through the collection of fossils geological processes into limestone (Hiskia and Tupamahu, 2001).

Carbon content contained in these rocks be the deciding factor in determining the age of a sample. Carbon is what is referred to as radiocarbon. Radiocarbon withdrawal by Faure (1986) in Siregar (2011) is the radiometric method that can be used to determine the absolute age of a sample by age 50,000 years ago. Method of age determination can only be made on materials that contain the element carbon. Elemental carbon isotope ¹⁴C used is contained in atmospheric CO₂ bound in a compound. ¹⁴C isotope is produced by the reaction of cosmic rays with nitrogen.

\[ \_6^0n + \_7^{14}N \rightarrow \_6^{14}C + \_1^1p \]

Isotope is called radiocarbon. Carbon isotope widely distributed in nature and found in every organic compound.

One method that is very popular in determination age of an object is a method of LSC (Liquid Scintillation Counting). This method works trace radioisotopes, in particular β emitting isotopes on the basis of the interaction of a solution of organic compounds that can interact with radiation (Salam, 1993).

The principle of the liquid scintillation counting method, samples containing radionuclides dissolved or suspended in a solution of scintillator (cocktail) that fits inside the glass or plastic vial. Radioactive particles in the sample is dissolved in a scintillator solution will collide with molecules of the solvent causes solvent molecules become excited and led to the scintillator molecules emit photons. The photons then detected by the PMT, so that the resulting electrical pulses are proportional to the energy of radioactive particles (Tjahaja and Mutiah, 2000).

Liquid scintillation counting method is usually only used for the enumeration of β low-energy radiation, such as ³H and ¹⁴C, but with the development of a liquid scintillation counter instrument, this method can be used also for the enumeration of total α and β. Advantages compared with the previous method is a liquid dosage form so as to facilitate sample homogeneously soluble so there is no effect of self-absorption and more efficient sample detection time (Tjahaja and Mutiah, 2000).

**METHODS**

**Time and Location**

This study conducted in October-December 2013 in the Radiation Chemistry Laboratory, Department of Chemistry, Faculty
of Mathematics and Natural Sciences, University of Hasanuddin, Makassar.

**Tools and Materials**

Hammers, files, LSC (Liquid Scintillation Counter) Hidex 300 SL and limestone samples from Leang-Leang Cave.

**Methods**

**Sampling Limestone Rock**

Limestone samples were taken using a file and a hammer at two point coordinates. Then, pieces of limestone rocks inserted into the plastic to be brought into the laboratory.

**Washing Limestone Rock Samples**

Limestone samples washing done in two stages, such as:

1. **Physical wash**

   Limestone samples cleaned with water and brushed and followed by rinse. After that, placed in the cup and allowed to stand a few days for the drying process. Limestone that has been dried and then broken into small pieces and weighing is done first to determine the initial weight.

2. **Chemical leaching**

   Washing step is then performed by immersing samples of limestone that had been weighed in a mixed 50/50 30% H₂O₂ and 1 N NaOH in a 100 mL beaker is placed in an ultrasonic for ±10 minutes and proceed with the separation of wash solution, then rinsed with distilled water several times until the rest of the wash solution is lost. The next stage, limestone samples immersed back into the mix 50/50 30% H₂O₂ and 1 N HClO₄ in a 100 mL beaker for ±30 seconds, and then separated again from the wash solution. The last stage of the washing process is by soaking the samples in 6 N HCl solution for 15-60 seconds and separated again from the wash solution, then rinsed again limestone samples with distilled water several times. Furthermore, limestone is dried in an oven at a temperature of 60 °C until the dry rocks. After that, weighed again to determine the weight % limestone lost during the washing process takes place.

**Analysis of CO₂**

Each rock sample that has been dried and finely crushed to 10 grams were taken for analysis of ¹⁴C or carbon content of the total. 10 gram samples that have been smoothed placed in a round bottom flask, then rinsed thoroughly with a solution of 85 % H₃PO₄ were placed in a separating funnel until all the powder samples completely reacted with H₃PO₄. Then, the resulting CO₂ gas is passed into a solution of 10 % KOH, as shown in Figure 1.

![Figure 1. Design tools as CO₂ separation carbonate limestone sample.](image)

**Determination of Total Carbon**

Solution of K₂CO₃ dissolved pipetted 10 mL into erlenmeyer for titration with 5 M
Leang cave Maros, South Sulawesi Province. This location is one of the protected historic attractions and are within the scope of Maros, especially at Leang - Leang village. Sampling is done on two limestone certain point. The selection of this location as a place of sampling because the limestone Leang-Leang Cave is one of the heritage assets within 20 km of the town of Maros or 44 Km from the city of Makassar and can be reached for ± 1 hour drive. There are two limestone samples taken from the study site. Both samples of limestone can be seen in Figure 2 below.

**Enumeration Limestone Rock Samples**

Enumeration of the samples was done by 8 mL of sample solution is pipetted into a glass vial of 20 mL, then added 12 mL of scintillator and shaken until homogeneous. Furthermore, the sample with the LSC chopped Hidex 300 SL made with interval time 1-300 minutes.

**Calculation of Absolute Age**

Age calculation is done using the formula:

\[
\begin{align*}
\frac{A}{A_o} &= \frac{t_{1/2}}{ln\frac{1}{2}} \ln \frac{A_o}{A} \\
A &= \text{Radioactivity of } ^{14}\text{C isotopes in the sample} \\
A_o &= \text{Radioactivity isotope } ^{14}\text{C during the life of plants or animals 15,3 DPM (Libby, 1960)} \\
t_{1/2} &= \text{Half-life of } ^{14}\text{C 5730 years} \\
ln2 &= 0,693
\end{align*}
\]

**RESULTS AND DISCUSSION**

**Sampling Location**

Sampling sites located in the limestone Leang-
or to destruction and leaching

the decay of radioactivity will decrease contin-

uously.

Cleaning Sample

Pretreatment samples of limestone are

physically laundering. Washing is done in stag-

es beginning with the washing with water fol-

lowed by washing with distilled water is used

to remove impurities that are easily lost as soil.
The next stage is a chemical leaching is by im-

mersing samples of limestone into a solution of

30% H₂O₂ and 1 N NaOH (50/50) in a 100 mL

beaker was placed in an ultrasonic apparatus

for 10 minutes to speed up the washing process

by giving vibrations the base and walls of the

sample container during the washing process

takes place. Meanwhile, wash with a solution

of 30 % H₂O₂ and HClO₄ 1 % (50/50) in a 100

mL beaker for ± 30 seconds intended to oxi-
dize other pollutant sources are not lost during

washing with 1 N NaOH solution and elimi-
nate the dyes contained limestone rock sam-

ples to be pure white. Last laundering using

chemicals is with 6 N HCl which serves to

eliminate impurities and secondary carbon

sources from the sample surface which has not

been lost on the previous washing. Washing

with 6 N HCl is done during 30-60 seconds.
The missing part of the sample is a natural

contamination accumulated over limestone in

contact with air and surrounding environment.
Results of treatment of the samples is shown in

Table 3.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Coordinate Point</th>
<th>Washing Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Physics (g)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Chemistry (g)</td>
</tr>
<tr>
<td>BG I</td>
<td>04°39’37,4”</td>
<td>87,956</td>
</tr>
<tr>
<td></td>
<td>119°27’14,6”</td>
<td>85,000</td>
</tr>
<tr>
<td>BG II</td>
<td>04°39’41,8”</td>
<td>80,176</td>
</tr>
<tr>
<td></td>
<td>119°27’21,9”</td>
<td>76,617</td>
</tr>
</tbody>
</table>

Weighing the two limestone samples

that BG I and BG II is done after the sample

was dried. Based on data from the weighing

results as shown in Table 3 above, the differ-

ence in initial weight gained weight after

weighing the sample for each sample limestone

BG I 2,956 grams, while the limestone samples

BG II 3,559 grams. The weight difference in

the two samples is the number of sample

weight lost during the washing is done , which

is about 3,36 % limestone samples BG I and

4,44 % for limestone samples BG II of weight

initially. The results of this washing produce

sample weight lost is not much different from

the one described by Adkins et al, (2002) that

the process of washing the sample with chemi-
cal compounds such as the above can eliminate

the weight of the samples ranged from 5-10 %

by weight before. That is limestone samples

have small amounts of impurity components

when compared to other environmental sam-

ples that have a number of small pores on its

surface.

Analysis of CO₂

Chemical components into the main

constituent in limestone is CaCO₃. Carbonate

contained in the limestone can be separated by

treatment with 85 % H₃PO₄. This step can be

done after the sample has been crushed lime-

stone to be smooth, so that the contact area be-

tween H₃PO₄ 85 % with the sample becomes
more widespread and the reaction can take place quickly. The reaction that occurs when a sample of limestone was added with 85% H$_3$PO$_4$ slowly will produce gas bubbles on the surface of the sample, as shown in the reaction below.

\[
\text{CaCO}_3(s) + \text{H}_3\text{PO}_4(l) \rightarrow \text{CaHPO}_4(aq) + \text{CO}_2(g) + \text{H}_2\text{O}(l)
\]

The gas bubbles are released and the CO$_2$ gas is passed into a solution of 1 M KOH absorbent. In this process, not all the CO$_2$ gas is passed into a solution of 1 M KOH absorber capable arrested. This can be seen from the presence of CO$_2$ gas bubbles through the surface of the absorbent solution. CO$_2$ is absorbed by the absorbent solution of K$_2$CO$_3$ dissolved KOH will produce.

\[
\text{CO}_2(g) + \text{KOH}(l) \rightarrow \text{K}_2\text{CO}_3(l) + \text{H}_2\text{O}(l)
\]

K$_2$CO$_3$ solution is a solution of the sample to be measured by LSC (Liquid Scintillation Counter) to determine the amount of $^{14}$C activity.

**Determination of Total Carbon**

Determination of total carbon contained in the limestone samples can be done by titration. This method is very supportive in this study. The total mass of the carbon samples obtained from BG I as much as 0.6144 grams, whereas for sample BG II as 0.9696 grams. According Djuhariningrum and Rusmadi (2004) that the carbon content in weight of CaCO$_3$ that there is 12% carbon by weight. Calculation of the total carbon in limestone samples need to be done to get the value of the specific activity of $^{14}$C contained in the sample. The specific activity of $^{14}$C itself is expressed in units of disintegrations per unit mass of carbon (DPM/gC).

**Sample Enumeration**

The specialty tools Hidex LSC 300 SL when compared to other tools is a device enumerator MikroWin 2000 software system that is able to provide results of its absolute chopped. Enumerators sample solution is performed using a set of tools LSC (Liquid Scintillation Counter) Hidex 300 SL has an important role in the detection of $\beta$ particles emitted from the radioisotope $^{14}$C originating from the sample. Sample enumeration results with these tools will get the amount of $^{14}$C activity is expressed in units Disintegrations per Minute (DPM) and the results obtained during enumeration is done in units of counts Counts per Minute (CPM). This enumeration method works trace radioisotopes, in particular $\beta$ emitting isotopes on the basis of the interaction of a solution of organic compounds that can interact with radiation (Salam, 1993).

Enumeration can be done after the addition of 12 mL of scintillator solution in 8 mL of sample solution in a 20 mL vial. This process is carried out in poorly lit room to avoid contamination of free air containing CO$_2$. Then, enumerated in the span of 1-300 minutes. Scintillator solution used contains two components, namely primary and secondary solvent. Radioactive particles contained in the sample is dissolved in a scintillator solution will collide with molecules of the solvent so that the solvent molecules become excited and emit photons. The photons then detected by the PMT, so that the resulting electrical pulses are proportional to the energy of radioactive particles. According Satrio (2009) This method is done by counting the CO$_2$ absorbed by the absorbent solution, so that the activity of samples containing $^{14}$C in CO$_2$ directly enumerated. Analysis of samples by this method involves the scintillator solution that will collide with the solvent molecules to excited. At this point the energy is released in the form of photons or
light flicker. The flicker of light has a specific wavelength and when it comes to fotokatode layers in PMT (*Photo Multiplier Tube*) will release electrons from the layer. This will be multiplied by the electron dinode dinode contained in the PMT and eventually these electrons will be collected on anode in the form of electrical pulses. Data results of the second sample enumeration limestone can be seen in Table 2 below.

Table 2. The data sample enumeration limestone BG I and BG II with a span of 1-300 minutes

<table>
<thead>
<tr>
<th>No</th>
<th>Times (Minutes)</th>
<th>BG I Activity</th>
<th>BG II Activity</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>CPMs</td>
<td>TDCRs</td>
</tr>
<tr>
<td>1</td>
<td>1</td>
<td>224,520</td>
<td>0.824</td>
</tr>
<tr>
<td>2</td>
<td>15</td>
<td>221,820</td>
<td>0.740</td>
</tr>
<tr>
<td>3</td>
<td>30</td>
<td>215,850</td>
<td>0.816</td>
</tr>
<tr>
<td>4</td>
<td>60</td>
<td>217,020</td>
<td>0.853</td>
</tr>
<tr>
<td>5</td>
<td>120</td>
<td>218,600</td>
<td>0.779</td>
</tr>
<tr>
<td>6</td>
<td>180</td>
<td>221,550</td>
<td>0.724</td>
</tr>
<tr>
<td>7</td>
<td>240</td>
<td>220,010</td>
<td>0.731</td>
</tr>
<tr>
<td>8</td>
<td>300</td>
<td>219,769</td>
<td>0.853</td>
</tr>
<tr>
<td></td>
<td>Average</td>
<td>219,892</td>
<td>0.790</td>
</tr>
</tbody>
</table>

Based on Table 2 above shows the results of counts on which the shredded three minutes for 30 minutes for the first BG sample count values decreased from the previous chopped, shredded and then further increased. Increasing the results of counts due to the effect of the instability of the phase between the sample solution with a solution of K₂CO₃ dissolved scintillator is not homogeneous, so the results of counts that fluctuate between 215,850 to 224,520 CPM with an average of 219,892 chopped CPM. The results of counts if made in the shape of the graph will look like in Figure 3.

Figure 3.

Graph showing the relationship of CPM results limestone samples BG I against time.
Likewise with BG II samples have initial count values are very high and decreased in the fourth when chopped for 60 minutes. Similarly, the sample BG I, BG II sample enumeration results also fluctuated between 317,840 to 334,640 CPM with average chopped 322,778 CPM. The results of counts if made in the shape of the graph will look like in Figure 4.

![Figure 4](image)

**Figure 4.** Graph showing the relationship of CPM results limestone samples BG II against time chopped.

Although both samples above show the results with values berfluaktuasi chopped or rising slowly, it does not mean cacahannya value will remain constant over the average value of counts. This can be seen in the re-enumeration of each sample along with chopped blank BG contained in Table 3 and Table 4.

<table>
<thead>
<tr>
<th>No</th>
<th>Times (minutes)</th>
<th>BG I Activity CPM</th>
<th>TDCR</th>
<th>Blank Activity CPM</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>30</td>
<td>214,310</td>
<td>0.827</td>
<td>213,810</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td>218,280</td>
<td>0.844</td>
<td>216,020</td>
</tr>
<tr>
<td>3</td>
<td></td>
<td>215,210</td>
<td>0.822</td>
<td>215,120</td>
</tr>
<tr>
<td>4</td>
<td></td>
<td>219,250</td>
<td>0.848</td>
<td>217,420</td>
</tr>
<tr>
<td>5</td>
<td></td>
<td>215,650</td>
<td>0.841</td>
<td>215,610</td>
</tr>
<tr>
<td>6</td>
<td></td>
<td>217,420</td>
<td>0.849</td>
<td>216,210</td>
</tr>
<tr>
<td>7</td>
<td></td>
<td>217,250</td>
<td>0.852</td>
<td>217,210</td>
</tr>
<tr>
<td>8</td>
<td></td>
<td>214,680</td>
<td>0.852</td>
<td>214,110</td>
</tr>
<tr>
<td>9</td>
<td></td>
<td>219,820</td>
<td>0.857</td>
<td>219,720</td>
</tr>
<tr>
<td>10</td>
<td></td>
<td>219,950</td>
<td>0.857</td>
<td>218,210</td>
</tr>
<tr>
<td></td>
<td>Average</td>
<td>217,182</td>
<td>0.846</td>
<td>216,344</td>
</tr>
</tbody>
</table>

**Table 3.**
The data enumeration BG I and blank samples for 30 minutes 10 times repetition
While the results of counts blank that serves as a correction factor to the results of chopped samples gave an average yield of 216,344 CPM chopped smaller than chopped sample results. This means that the digital samples with a blank has an average increment of counts of 0.838 CPM.

**Table 4.** The data sample enumeration BG II for 60 minutes 10 times repetition

<table>
<thead>
<tr>
<th>No</th>
<th>Times (minutes)</th>
<th>BG II Activity</th>
<th>Blank Activity</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>CPM</td>
<td>TDCR</td>
</tr>
<tr>
<td>1.</td>
<td>60</td>
<td>314,640</td>
<td>0.888</td>
</tr>
<tr>
<td>2.</td>
<td></td>
<td>298,040</td>
<td>0.879</td>
</tr>
<tr>
<td>3.</td>
<td></td>
<td>301,740</td>
<td>0.883</td>
</tr>
<tr>
<td>4.</td>
<td></td>
<td>309,140</td>
<td>0.887</td>
</tr>
<tr>
<td>5.</td>
<td></td>
<td>305,840</td>
<td>0.901</td>
</tr>
<tr>
<td>6.</td>
<td></td>
<td>308,740</td>
<td>0.876</td>
</tr>
<tr>
<td>7.</td>
<td></td>
<td>314,700</td>
<td>0.908</td>
</tr>
<tr>
<td>8</td>
<td></td>
<td>311,740</td>
<td>0.880</td>
</tr>
<tr>
<td>9</td>
<td></td>
<td>308,430</td>
<td>0.828</td>
</tr>
<tr>
<td>10</td>
<td></td>
<td>313,760</td>
<td>0.722</td>
</tr>
<tr>
<td></td>
<td>Average</td>
<td>308,677</td>
<td>0.865</td>
</tr>
</tbody>
</table>

While the limestone samples for enumeration BG II as shown in Table 4 above, the results of chopped has variation although long used the same time. Counts range between 298,040-314,700 CPM which has an average of counts per minute 308,677 CPM with approximately 86.5 % counting efficiency. It is not much different from that described by Zhu and Yang (1995) that the enumeration for energy efficiency β ranged from 80-85%.

This can be explained by assuming that the decay is a statistical event. This suggests that the statistical properties of the atoms that will decay in the next second can not be ascertained because only a probability (Syahrir, 2001). Counting efficiency may be reduced because of the symptoms of burnout (quenching) which occurs due to the presence of oxygen or impurities in the solvent container scintillation vials. Blackout effect on β spectrum is the fluorescence spectrum shifts toward lower energy. The larger outage occurs, the fluorescence spectrum increasingly shifted to the left or toward the lower energy (Yariantto et al, 2001).
The use of blank enumeration herein is intended to determine the contribution of radiation from liquid scintillation counting environments that are not sampled. Results of counts blank is also used as a correction factor to the digital samples.

**Determination of Specific Activity Sample**

The specific activity of limestone samples can be determined from the difference between the results of counts Counts Per Minute (CPM) against the results of counts of blank samples generated as a correction factor to the outcome of chopped samples divided by the

<table>
<thead>
<tr>
<th>Samples</th>
<th>DPM</th>
<th>C-total (g)</th>
<th>As (DPM/gC)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BG I</td>
<td>0.989</td>
<td>0.6144</td>
<td>1.609 ± 0.0359</td>
</tr>
<tr>
<td>BG II</td>
<td>7.484</td>
<td>0.9696</td>
<td>7.718 ± 0.109</td>
</tr>
</tbody>
</table>

Based on data enumeration sample results in Table 5 above shows the specific activity of \(^{14}\text{C}\) in samples of limestone BG I is 1.609 ± 0.0359 DPM/gC, and samples of limestone BG II 7.718 ± 0.109 DPM/gC. Activity obtained indicates the magnitude of the decay of carbon atoms that take place every minute (DPM) per gram of carbon. Both the specific activity data obtained samples showed a very large difference between the two and is lower than the value of the average specific activity of modern living plants as the result of research conducted by Libby (1960) is often used as an early activity in the determination of the age of the sample environment ie 15.3 DPM/gC.

**Age Determination of Limestone Rock Samples**

Age of limestone samples can be determined by converting the measurement results obtained by the specific activity of each sample limestone into equation calculation of absolute age above.

From equation above obtained the second age limestone Leang-Leang cave constituent, as found in Table 6.
Table 6. The data on the calculation of age limestone through the measurement of $^{14}$C activity

<table>
<thead>
<tr>
<th>Samples</th>
<th>Age (years)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Limestone (BG I)</td>
<td>$25,607,403 \pm 919,305$</td>
</tr>
<tr>
<td>Limestone (BG II)</td>
<td>$8,457,792 \pm 921,899$</td>
</tr>
</tbody>
</table>

Calculation of $^{14}$C activity in the determination of the age of two limestone samples by liquid scintillation method gives results for each sample age limestone BG I $25,607,403 \pm 919,305$ years, while the limestone samples BG II $8,457,792 \pm 921,899$ years. As has been discussed earlier that BG I limestone samples are samples that have been detached from the wall of the cave and have mossy physically older than BG II the limestone samples were still strongly attached to the wall of the cave and the relatively younger, so the way from using hammer and miserly. Both age limestone samples were counted is the age of the sample length is dead or no longer live shows activity that counted before 2013. As for knowing when the sample is dead, then the age of death decreased by the length of the sample calendar year since it began in the 2013.

**CONCLUSION**

Age of limestone samples BG I calculated based on data specific activity of $^{14}$C is $25,607,403 \pm 919,305$ years, while the age of the rock sample limestone BG II is younger, which is $8,457,792 \pm 921,899$ years.

**REFERENCES**


