Development of $k_0$-INAA standardization method by neutron activation with Am–Be source

Huynh T. Phuong *, Mai V. Nhon, Luu D.H. Oanh

Department of Nuclear Physics, University of Science, VNU-HCMC, 227 Nguyen Van Cu Street, District 05, HCM City, Viet Nam

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A B S T R A C T

The $k_0$-standardization method on the $^{241}$Am–Be isotopic neutron source has been studied and developed. In this paper, the characteristics of the neutron spectrum in an isotropic neutron field of $^{241}$Am–Be isotopic neutron sources was experimentally determined by dual monitor method using measured cadmium ratios of $^{197}$Au($n, \gamma$)$^{198}$Au and $^{98}$Mo($n, \gamma$)$^{99}$Mo($\beta^-$)$^{99m}$Tc reactions. Application of the $k_0$-standardization method for the determination of elemental concentrations such as Al, Mn and Na in cement is carried out as well. The obtained results are in agreement with other analyses.

1. Introduction

The $k_0$-standardization method in neutron activation analysis on nuclear reactor was proposed in 1975 by A. Simonits (Simonits and De Corte, 1975). After that, in 1987, Frans De Corte studied developed in details (De Corte, 1987). The advantages of the $k_0$-standardization method are measuring multi-element without using reference materials, flexibility in experiment and high accuracy. Because of these advantages the $k_0$-standardization method is used extensively in nuclear reactors over the world. However, the $k_0$-standardization method has not been studied for neutron activation analysis on Am–Be isotopic sources yet. In 1994, an INAA facility was launched at the Department of Nuclear Physics (DNP), University of Science, Vietnam National University of Ho Chi Minh City (VNU–HCMC). It included $^{241}$Am–Be neutron source and Geiger–Müller counter. For this facility, it is mainly used to determine aluminum (Al) contents in bauxite ores (Tri, 1996). In 1998, Geiger–Müller counter was changed by a gamma-ray spectroscopy with HPGe detector. In 2004, a gamma-ray spectroscopy with HPGe detector has been installed. Because of high sensitivity of HPGe detector, the elemental concentration as Al, V, Mn, Cr, Cu, Zn, Br, As, Sb, Mo, Se, Au, etc., could be detected and measured in this facility. The disadvantages in INAA conventions are measuring the multi-element, and difficulty in preparation of standard samples. Hence, the development of $k_0$-standardization method is necessary for education and training in the Universities.

2. Methodology

In the $k_0$-standardization method of INAA, a sample with weight $w$ is irradiated by a neutron source at thermal neutron position, after irradiation the activities of sample are counted with gamma-ray spectroscopy, then the concentration $\rho_a$ of an analytic “a” is obtained from its measured isotope/gamma ray as (De Corte, 1987; De Corte et al., 2003)

$$\rho_a (\mu g/g) = \left( \frac{N_p}{w \times \text{DC}} \right) \times \frac{1}{k_0 \text{Au}(a)} \times \frac{G_{\text{th}Au} + G_{\text{th}Au} Q_{0 \text{Au}}(x)}{G_{\text{th}Au} + G_{\text{th}Au} Q_{0 \text{Au}}(2)} \times \frac{f_{\text{th}Au}}{f_{\text{th}Au}} \times 10^6$$

where “Au” refers to the co-irradiated gold monitor [$^{197}$Au($n, \gamma$)$^{198}$Au, $E_\gamma$ = 411.8 keV] and $N_p$ is the net number of counts in the full-energy peak (corrected for pulse losses), $W$ is the weight of the gold monitor, $w$ is the weight of sample, $t_m$ is the measuring time, $S = 1 - \exp(-\lambda t_i)$, $t_i$ is the irradiation time, $D = \exp(-\lambda t_d)$, $t_d$ is the decay time, $C = [1 - \exp(-\lambda t_m)]/\lambda t_m$, $\lambda$ is the decay constant, $f$ is the thermal to epithermal neutron flux ratio, $Q_{0 \text{Au}} = k_0 \sigma_a$ (resonance integral to 2200 ms$^{-1}$ cross-section ratio), $\sigma_a$ is the measure for the epithermal neutron flux distribution, approximated by a 1/1$+\delta$ dependence ($\delta$ is considered to be independent of neutron energy), $f_{\text{th}Au}$ is the full-energy peak detection efficiency, $G_{\text{th}Au}$ and $G_{\text{th}Au}$ are factors for correction of thermal and epithermal neutron self-shielding, respectively.

In Eq. (1), $k_0\text{Au}(a)$ is defined as

$$k_0\text{Au}(a) = \frac{M_{\text{Au}} \theta a_0 \sigma_a} {M_{\text{Au}} \theta a_0 \sigma_a + A_{\gamma}}$$

where, $M$ is the atomic weight, $\theta$ is the isotopic abundance, $a_0$ is the 2200 ms$^{-1}$ ($n, \gamma$) cross-section, and $\gamma$ is the absolute gamma-ray intensity (emission probability).
The $k_{0,\text{Au}}$ in the above Eq. (2) is a constant for nuclear composition and experimentally measured and tabulated in reference literatures (De Corte, 1987; De Corte et al., 2003). Hence, $k_{0,\text{Au}}$ of the isotopes is independent with neutron spectrum, and it can be applied to any neutron source.

Generally, for the development of $k_0$-standardization method on INAA system, the measurements are needed to be carried out as follows:

- For the neutron source spectrum: the characteristics of the neutron spectrum, such as the thermal to the epithermal neutron flux ratio ($f$) and the epithermal neutron flux shape factor ($\alpha$), are experimentally determined.
- For the gamma-ray spectroscopy: the curves of detector efficiency are experimentally measured at counting positions.
- And the net count at full-energy peak is corrected for pulse losses.

3. Experiments

3.1. Instrumental neutron activation analysis system

In this work, an INAA system includes Am–Be neutron source with SCi activity ($\sim 1.5 \times 10^7$ ns$^{-1}$ in intensity), a gamma-ray spectroscopy with HPGe detector and an automatic unit used to transfer the sample from the irradiation hole to counting chamber and vice versa.

The Am–Be neutron source was produced by mixing $^{241}$Am radioisotope with BeO powder. Produced neutron with 4.7 MeV average energy in reaction as

$$^{241}\text{Am} \rightarrow \alpha + ^{9}\text{Be} \rightarrow ^{12}\text{C} + ^{1}\text{n}$$

The Am–Be source configuration is shown in Fig. 1. It includes two channels for neutron irradiation: one channel is used for fast neutron irradiation (called fast channel) and the other one is used for thermal neutron irradiation (called thermal channel). The surrounding of neutron source is covered by blocks of paraffin to get the thermal neutron at thermal channel, and the outer is shielded by lead. This neutron source is underground and a concrete wall of 50 cm thickness is used to separate neutron source and control room.

The sample is irradiated at thermal neutron channel or/and fast neutron channel by an automatic sample transfer system, which is controlled by a computer program as shown in Fig. 2.

After neutron irradiation, sample is transferred to the counting chamber of gamma-ray spectroscopy, which was 1.8 keV energy resolution at 1332 keV peak of $^{60}$Co. The net count of full-peak energy is estimated by Genie-2k software. The gamma-ray spectroscopy is shown in Fig. 3.

3.2. Experimental determination of detector efficiency curves

3.2.1. For cylinder samples

In this work, a reference material namely IAEA-375, which contained radionuclides and trace elements in soil, is contained in the polyethylene cylinder box, which was 4.5 cm in height and 1.5 cm in diameter. Reference material of 5.43 g, was put horizontally and distance of 1 cm to the detector surface and counted in 48 h. The net count of full-energy peaks as 63.02 keV ($^{234}$Th), 77.1 keV ($^{212}$Pb), 238.6 keV ($^{212}$Pb), 351.1 keV ($^{211}$Bi), 609.3 keV ($^{214}$Bi), 661.7 keV ($^{137}$Cs), 911.6 keV ($^{228}$Ac) and 1460.8 keV ($^{40}$K) were estimated by the Genie-2k software. After that, efficiency of the detector was determined, and a curve was fitted with third-order function as shown in Fig. 4. All of the measurements are corrected for true-coincidence effects. In the previous work, a computer program (named COIPro) was written by C# language, which is used to correct true-coincidence effects (Phuong, 2010). It is based on the formulas of Frans De Corte (De Corte, 1987).

3.2.2. For foil samples

At the INAA laboratory of DNP, the shapes of samples are not only cylindrical but foil as well. To avoid errors in sample geometries, it is necessary to determine the curve of detector efficiency using the standard radioisotope sources with foil geometry. In this work, standard radioisotope source in foil shape was done by irradiating Indium foils (purity of 99.9%), with 1 mm in thickness and weight of 64.3 mg, at the thermal neutron channel in 1 h, and nuclear reaction was produced $^{115}\text{In}$ ($n, \gamma$)$^{116}\text{mIn}$, $T_{1/2}=54.3$ min. Foil’s activities at the end of the
irradiation is as follows:

$$A(Bq) = \frac{w \cdot \theta \cdot N_A}{M} \cdot \sigma_0 \cdot \phi \cdot (1 - e^{-\lambda t_i})$$

(3)

where, $w$ is the weight of foil, $\theta$ is the isotopic abundance, $N_A$ is Avogadro’s constant, $M$ is the atomic mass, $\sigma_0$ is the cross-section at $v_0 = 2200$ ms$^{-1}$, $\phi$ is the thermal neutron flux, $\lambda$ is the decay constant, and $t_i$ is the irradiation time.

As a result, $A(Bq) = (15.61 \pm 2.30)$ is the activity, which was obtained at the end of the irradiation time. This foil is put at 1 cm distance from the detector surface and counted in 1 h. The area of full-energy peaks of the $^{110m}$In radioisotope such as 138.32 keV, 416.86 keV, 818.72 keV, 1097.33 keV and 1293.56 keV were calculated by the Genie-2k software, then detector efficiency of full-energy peaks was determined, and it was fitted with linear function as shown in Fig. 5. All of measurements are corrected for true-coincidence effects. In the previous work, a computer program (named COIPro) was written by C# language, which used to correct the true-coincidence effects (Phuong, 2010). It is based on the formulas of Frans De Corte (De Corte, 1987).

3.3. Experimental determination of neutron spectrum parameters of Am–Be source

3.3.1. Experimental determination of $\alpha$ and $f$ factors

In this experiment, the method of dual-monitor cadmium ratio was used to measure $\alpha$ and $f$ factors. Two monitors used in this work were gold ($^{197}$Au) and molybdenum ($^{98}$Mo), and they are irradiated with and without Cd-covered.

At the thermal neutron channel of Am–Be source, an Au foil of 50 mg (purity is 70%) was irradiated with and without Cd-covered foil (1 mm thickness) in 24 h and 48 h, respectively. Meanwhile, the Mo monitor was prepared in the form of MoO$_3$ powder with weight of 240 mg and contained in a small plastic package. After that, Au foil and MoO$_3$ powder irradiated with and without Cd-covered in 24 h and 48 h, respectively. After neutron irradiation, the monitors are transferred to counting chamber of HPGe detector and counted in 24 h and 48 h, according to measured monitor. The specified activities of monitors were calculated as follows:

$$A_{sp} = \frac{N_p}{w_{SDC}}$$

(4)

where, $N_p$ is the net count of full-energy peak during counting time, $w$ is the weight of monitor; $C = 1 - e^{-\lambda t_i}$, $t_i$ is the irradiation time, $\lambda$ is the decay constant; $D = e^{-\lambda t_d}$, $t_d$ is the decay time and $C = (1 - e^{-\lambda t_d})/\lambda t_d$, $t_m$ is the counting time.

The Cd ratio was defined as

$$R = \frac{A_{sp}}{A_{sp}/F_{Cd}}$$

(5)

where, $A_{sp}$ and $A_{sp}$ are specified activities of the monitor with and without Cd-covered, respectively; $F_{Cd}$ is the cadmium transmission factor for epithermal neutrons.

Hence, the epithermal neutron flux shape factor, $\alpha$, determined from equation

$$\frac{(R-1)_{Au}}{(R-1)_{Mo}} = \frac{[Q_0 - 0.429]_{Mo}}{[Q_0 - 0.429]_{Au}} \cdot \frac{[E_{f, Mo}]}{[E_{f, Au}]} \cdot C_\alpha = 0$$

(6)

with

$$C_\alpha = \frac{0.429}{(2\alpha + 1) \times 0.55^2}$$

(7)

The nuclear data of Au and Mo monitors used in this work are shown in Tables 1 and 2. The obtained values of Au and Mo monitors in this experiment were combined with nuclear data from Tables 1 and 2 and replaced into Eqs. (5) and (6). The resulting Cd-ratio ($R$) and the epithermal neutron flux shape factor ($\alpha$) were determined, as shown in Table 5.

<table>
<thead>
<tr>
<th>Reaction</th>
<th>Half time (h)</th>
<th>Measured gamma ray energies</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Gamma ray energy, $E$ (keV)</td>
</tr>
<tr>
<td>$^{98}$Mo($n, \gamma$)$^{99}$Mo</td>
<td>65.94</td>
<td>140.511</td>
</tr>
<tr>
<td>$^{98}$Mo($n, \gamma$)$^{99}$Mo($\beta$, $^{99}$Mo)</td>
<td>6.015</td>
<td>140.511</td>
</tr>
<tr>
<td>$^{197}$Au($n, \gamma$)$^{198}$Au</td>
<td>64.684</td>
<td>411.800</td>
</tr>
</tbody>
</table>

Fig. 4. Curve of detector efficiency for cylinder sample.

Fig. 5. Linear detector efficiency for foil sample.
When the \( \alpha \) factor was determined from Eq. (6), the \( f \) factor was determined as follows:

\[
f = (F_{Cd}R - 1)Q_0(\alpha)/G_n
\]  

(8)

with,

\[
Q_0(\alpha) = \frac{Q_0}{(E_f)^{\alpha}} + \frac{0.429}{(2x+1) \times 0.55^2}
\]  

(9)

By replacing the parameters of nuclear data in Tables 1 and 2 into Eqs. (5), (8) and (9), the \( f \) factor was determined as shown in Table 3. In this work, thermal and epithermal neutron self-shielding, \( G_n \) and \( G_e \) in Au and Mo monitors were calculated (Phuong et al., 2010).

### Table 2

<table>
<thead>
<tr>
<th>Monitor</th>
<th>Reaction</th>
<th>( E_f (\text{eV}) )</th>
<th>( F_{Cd} )</th>
<th>( Q_0 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mo</td>
<td>( ^{198}\text{Mo}(n,\gamma)^{199}\text{Mo} )</td>
<td>211</td>
<td>1.0</td>
<td>53.2</td>
</tr>
<tr>
<td>Au</td>
<td>( ^{197}\text{Au}(n,\gamma)^{198}\text{Au} )</td>
<td>5.47</td>
<td>0.991</td>
<td>15.7</td>
</tr>
</tbody>
</table>

All of measurements are corrected for true-coincidence effects. In the previous work, a computer program (named COIPro) was written by C\# language, which was used to correct true-coincidence effects (Phuong, 2010). It is based on the formulas of Frans De Corte (De Corte, 1987).

#### 3.3.2. Experimental determination of thermal neutron flux \( \phi_{th} \) and epithermal neutron flux \( \phi_{e} \)

In this work, two monitors were carried out. The one is Au-wire (diameter of 1 mm), and the other one is Au-foil (thickness of 1 mm), with weight of 37.52 mg and 25.10 mg, respectively. The monitors are irradiated at thermal neutron channel in 48 h, and gamma-ray activities are counted at a close source-detector distance of 1 cm in 24 h. Full-energy 411.8 keV peak area of \( ^{199}\text{Au} \) was estimated using Genie-2k software. The thermal neutron flux is measured as follows:

\[
\phi_{th} = \frac{(N_p/t_m)M}{W_{\text{Au}p}(E_f)G_nG_m + G_0Q_0(\alpha)/f_{\text{SDC}}}
\]  

(10)

The parameters in Eq. (10) are shown in the previous sections.

By replacing the parameters of \( ^{199}\text{Au} (n,\gamma)^{198}\text{Au} \) reaction in Eq. (10), the thermal neutron flux is determined. And from measured \( f \) factor above, epithermal neutron flux is also determined as shown in Table 4. In this work, thermal and epithermal neutron self-shielding, \( G_n \) and \( G_e \) in Au and Mo monitors were calculated (Phuong et al., 2010).

#### 3.4. Application of \( k_0 \)-standardization method for determination of concentration of Na, Al and Mn in cement samples

In this work, five types of cement samples are done, which are denoted as VN1, VN2, THAI, CHI and INDO. Each type of sample is prepared with four samples, and the weight of sample is from 7 g to 8 g. The prepared sample is contained in a cylinder polyethylene box, which was 50 mm in height and 15 mm in diameter, and co-irradiated with a monitor of Au (70% in weight), which weighted 27 mg.

Sample is irradiated at the thermal neutron channel of the Am–Be neutron source in 15 min (for determination of Al) and 24 h (for determination of Na and Mn). After neutron irradiation, sample is transferred to counting chamber of HPGe detector, which 1.8 keV resolution at 1332 keV peak of \( ^{60}\text{Co} \), and is counted in 8 min (for determination of Al) and in 2 h (for determination of

### Table 4

<table>
<thead>
<tr>
<th>Monitor</th>
<th>Neutron flux (n cm(^{-2}) s(^{-1}))</th>
<th>Thermal neutron flux, ( \phi_{th} )</th>
<th>Epithermal neutron flux, ( \phi_{e} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Au (foil)</td>
<td>679.74 ± 15.20</td>
<td>26.42 ± 3.74</td>
<td></td>
</tr>
<tr>
<td>Au (wire)</td>
<td>658.56 ± 9.15</td>
<td>25.14 ± 3.04</td>
<td></td>
</tr>
<tr>
<td>Average</td>
<td>669.15 ± 17.88</td>
<td>25.78 ± 4.81</td>
<td></td>
</tr>
</tbody>
</table>

### Table 5

Concentration of elements of Na, Al and Mn in cement sample types.

<table>
<thead>
<tr>
<th>Samples name</th>
<th>Element</th>
<th>Concentration, ( \rho (%) )</th>
<th>( k_0 )-INAA method</th>
<th>INAA method</th>
<th>ICP-MS(^a)</th>
<th>ICP-MS(^b)</th>
</tr>
</thead>
<tbody>
<tr>
<td>VN1</td>
<td>Mn</td>
<td>0.057 ± 0.012</td>
<td>0.058 ± 0.005</td>
<td>0.050 ± 0.005</td>
<td>0.053 ± 0.003</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Na</td>
<td>0.239 ± 0.014</td>
<td>0.242 ± 0.032</td>
<td>0.220 ± 0.010</td>
<td>0.300 ± 0.040</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Al</td>
<td>3.965 ± 0.120</td>
<td>3.853 ± 0.150</td>
<td>3.730 ± 0.050</td>
<td>3.200 ± 0.030</td>
<td></td>
</tr>
<tr>
<td>VN2</td>
<td>Mn</td>
<td>0.056 ± 0.007</td>
<td>0.054 ± 0.005</td>
<td>–</td>
<td>–</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Na</td>
<td>0.397 ± 0.055</td>
<td>0.411 ± 0.046</td>
<td>–</td>
<td>–</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Al</td>
<td>2.984 ± 0.426</td>
<td>3.130 ± 0.450</td>
<td>–</td>
<td>–</td>
<td></td>
</tr>
<tr>
<td>THAI</td>
<td>Mn</td>
<td>0.055 ± 0.001</td>
<td>0.053 ± 0.002</td>
<td>–</td>
<td>–</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Na</td>
<td>0.240 ± 0.004</td>
<td>0.252 ± 0.022</td>
<td>–</td>
<td>–</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Al</td>
<td>1.792 ± 0.392</td>
<td>1.732 ± 0.252</td>
<td>–</td>
<td>–</td>
<td></td>
</tr>
<tr>
<td>CHI</td>
<td>Mn</td>
<td>0.038 ± 0.003</td>
<td>0.041 ± 0.005</td>
<td>–</td>
<td>–</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Na</td>
<td>0.132 ± 0.008</td>
<td>0.134 ± 0.010</td>
<td>–</td>
<td>–</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Al</td>
<td>2.846 ± 0.233</td>
<td>2.785 ± 0.050</td>
<td>–</td>
<td>–</td>
<td></td>
</tr>
<tr>
<td>INDO</td>
<td>Mn</td>
<td>0.055 ± 0.001</td>
<td>0.061 ± 0.005</td>
<td>–</td>
<td>–</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Na</td>
<td>0.530 ± 0.026</td>
<td>0.542 ± 0.032</td>
<td>–</td>
<td>–</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Al</td>
<td>1.005 ± 0.313</td>
<td>1.073 ± 0.253</td>
<td>–</td>
<td>–</td>
<td></td>
</tr>
</tbody>
</table>

\(^a\) Analytical and Testing center, Union of Geological Mapping of Southern Vietnam.

\(^b\) Analytical and Testing center, Department of Science and Technology in Ho Chi Minh City.
Na and Mn). Gamma-ray spectral of the sample is obtained by Genie-2k software. All of measurements are corrected for true-coincidence effects. In the previous work, a computer program (named COLPro) was written by C# language, which was used to correct true-coincidence effects (Phuong, 2010). It is based on formulas of Frans De Corte (De Corte, 1987).

By application of Eq. (1), the elemental concentration as Al, Na and Mn in five types of cement was determined as shown in Table 5. In there, elemental concentration in the VN1 sample is measured by the ICP-MS method. In this work, the elemental concentration as Al, Na and Mn in cement samples was also checked by the relative method in INAA technique. Here, a comparison sample is made by mixing 5%Al2O3, 1.5%MnO2 and 5%NaCl. Its experimental conditions are similar to that of the cement samples. Standard deviations were 1σ for all of measured values as shown in Table 5.

4. Results and discussions

Two main experiments in the development of k0-INAA standardization have been done on the Am–Be neutron source at DNP. In the first one, the efficiency of detector at the counting position, which is close to the surface of the detector, has been measured for two geometries of the sample. It included cylinder and foil samples. The efficiency of the detector for the cylinder sample is fitted to the third-order function of log(np) vs. log(ep)

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In the second one, the parameters of the neutron spectrum have been measured by dual monitor method with monitors as Au and Mo at thermal neutron channel using cadmium ratios of 197Au(n, γ)198Au and 98Mo(n, γ)99Mo(β–γ99mTc) reactions. In these experiments, peak of 140.5 keV of 99mTc was measured with correction of peak of 140.5 keV of 99Mo (Haluk et al., 2004) because 99mTc is the daughter of 99Mo. By the foil activation method, thermal neutron flux has been determined at the thermal neutron channel. It is clear that, although thermal and epithermal neutron fluxes were very small, but they are suitable in training and research at the Universities on neutron activation analysis as well as in other fields.

Application of k0-INAA standardization method has been carried out in the determination of concentration of elements as Al, Na and Mn in cement samples. The obtained values are in good agreement with the ICP-MS method, including Mn (within 12%), Na (within 8%) and Al (within 6%).

5. Conclusions

The k0-INAA standardization method on the isotope neutron source has been researched and developed at the DNP. As the result, the efficiency of the detector has been measured for two geometries of the sample at the counting position close to the surface of the detector, including cylinder and foil sample. The parameters of the neutron spectrum have been measured at the thermal neutron channel using cadmium ratios of 197Au(n, γ)198Au and 98Mo(n, γ)99Mo(β–γ99mTc) reactions. For the low flux, determination of elemental concentration is suitable for isotopes with large activation cross-section and short/medium half-life. The elements that could be analyzed on this facility include Al, V, Mn, Cr, Cu, Zn, Br, As, Sb, Mo, Se, Au, etc. This paper shows a principle of INAA based on the k0-standardization method using Am–Be neutron source, which is suitable for training and research at the DNP, University of Science, VNU-HCMC.

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