Experimental Examination

Answer All the Questions

(3.5 Hours)

SAFETY PRECAUTIONS

Thoroughly review the entire experimental procedure before commencing the experiment to understand the steps involved and identify potential hazards. Follow all safety instructions to minimize the risk of accidents.

1. It is required to always wear disposable nitrile gloves and lab coats when handling hazardous chemicals. Safety goggles and face masks should be worn if deemed necessary.
2. Inform the exam invigilator of any mishaps during chemical handling.
3. Familiarize yourselves with the potential hazards of the chemicals used in the experiment, including flammability and toxicity.
4. Locate the nearest fire extinguisher for use in case of an emergency.

REMINDERS

1. Write all your answers including steps / solutions on the answer sheet. You will not be allocated any time to write down your answers at the end of the exam.
2. Write your answers including steps / solutions using ink pens. Final answers must be written in ink to ensure clarity when the answer sheets are scanned. We also recommend retracing any graphs or illustrations with ink after sketching them in pencil, to make sure they are clearly visible in the scanned copy.
3. There is a box on your table containing the materials required for this experiment.

4. All waste related to the experiment should be disposed of into the containers provided.
5. All consumables prepared for each student should be sufficient for said experiment. If it's insufficient or missing, you may silently raise your hand.

The invigilator, if he/she judged the consumable was truly insufficient, will try to provide the extra consumables requested but it may take time and not be universally available.

E1 Effects of Gamma Irradiation on Stability of Ascorbic Acid (10 pts)

In this experiment, a 2, 6 sodium dichlorophenol indophenol (DCPIP) solution will be used to measure vitamin C concentrations (ascorbic acid) within a solution. Several solutions will be prepared, including a DCPIP solution, standard ascorbic acid solutions and sample ascorbic acid solutions. The ascorbic acid solutions used were irradiated at various radiation doses beforehand. Finally, A titration process will be performed onto the sample solutions using the DCPIP solution while the volume used will be recorded to determine the ascorbic acid content in each sample solution. Remember to go through and confirm that all items are provided and complete following the material list in **Given Materials**.

Write numerical answers with only two decimal places (rounding off) in the answer sheet. All numerical answers should be written with appropriate SI units.

Experimental Guide

Vitamins are organic compounds needed to maintain physiological functions and the well-being of the human body, where they act as coenzymes, antioxidants, and precursors for various biochemical processes. However, most vitamins cannot be synthesized by the body and are sourced from the diet. Since the levels of vitamins such as Vitamin A, B, C and D in our diets are usually already insufficient to meet daily recommended intake levels, any post-harvest treatment that affects the stability of vitamins will further deplete the nutritional value of the food.

Food irradiation is a widely recognized post-harvest technology used to enhance food safety, extend shelf life, and maintain quality. Gamma irradiation of agricultural produce serves several purposes. Firstly, this eliminates pathogenic microorganisms, such as *Salmonella* and *Escherichia coli (E. Coli)*, which reduces the incidence of foodborne illnesses. The removal of microorganisms also leads to spoilage prevention by inhibiting the main cause of spoilage and food decomposition. Fruits from tropical areas are also frequently irradiated as a form of pest control as opposed to chemical fumigation that may degrade the quality of the fruits. Additionally, gamma irradiation delays the sprouting of vegetables such as potatoes and the ripening of fruits, which contributes to prolonging their longevity and marketability. Moreover, high-dose radiation can be used to sterilize food products, which could lengthen their shelf life for years, making them suitable for patients with a compromised immune system.

Effects of Gamma Irradiation on Vitamin Stability

One of the primary concerns associated with food irradiation is its potential impact on the stability of micronutrients, particularly vitamins. Water-soluble vitamins such as ascorbic acid (Vitamin C) and certain B vitamins are particularly sensitive to ionizing radiation due to their chemical structures, rendering them susceptible to oxidative degradation. Vitamin C in particular is prone to decomposition when exposed to heat, oxygen and radiation.

Gamma irradiation may induce chemical modifications in food primarily through radiolysis, generating reactive oxygen species (ROS) and free radicals. The process begins with the oxidation of ascorbic acid to dehydroascorbic acid (DHA) via the loss of two electrons, facilitated by free radicals generated during gamma irradiation. DHA remains biologically active, but is unstable in aqueous solutions and readily hydrolyzes to form diketogulonic acid, an irreversible breakdown product that lacks vitamin activity. Additionally, further oxidative cleavage of ascorbic acid and DHA leads to the formation of smaller organic acids, such as oxalic acid, which contribute to nutritional losses. The rate of degradation is significantly influenced by environmental factors, including oxygen concentration, pH, and temperature. Higher oxygen availability accelerates the breakdown process, while acidic conditions tend to slow down oxidation. The presence of metal ions, such as iron and copper, can catalyze oxidation reactions, further exacerbating vitamin C loss. In the context of gamma irradiation, the dose-dependent increase in reactive oxygen species directly correlates with the extent of ascorbic acid degradation, highlighting the importance of optimizing irradiation conditions to preserve nutritional quality.

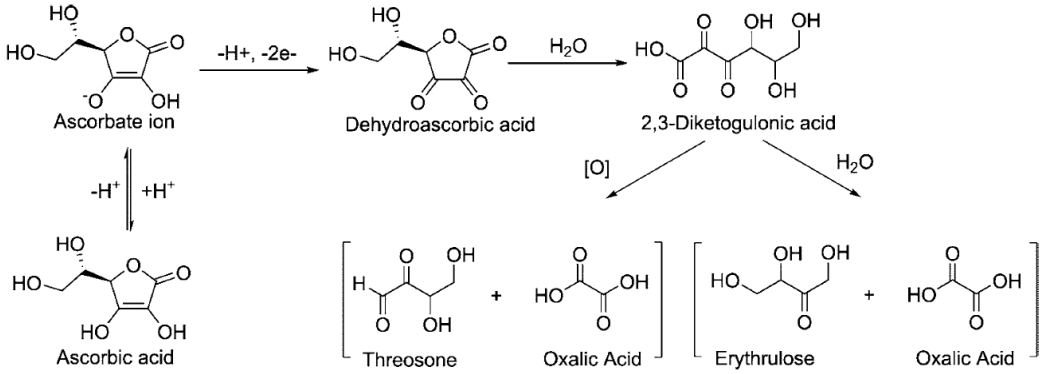

Figure E1.1. *Degradation pathways of ascorbic acid*

Applications and Implications

Understanding the effects of gamma irradiation on vitamin stability is crucial for optimizing food processing techniques that preserve nutritional value. If significant nutrient losses occur at specific doses, alternative post-harvest treatment strategies such as lower-dose irradiation combined with antioxidant fortification or modified packaging conditions may be explored to minimize degradation.

A broader discussion on the trade-off between food safety and nutritional retention in irradiated food products is also important, as it provides insights for policymakers and food regulatory authorities to develop guidelines that balance microbiological safety with nutrient preservation. The findings can thus be utilized in food labeling policies, granting customers the right to make informed decisions in the consumption of irradiated food. Furthermore, food manufacturers can utilize the findings to enhance product formulations, possibly incorporating protective antioxidants to counteract vitamin degradation. From a medical perspective, understanding the stability of ascorbic acid in irradiated foods is particularly important for populations with higher nutritional requirements, such as children, pregnant women, and individuals with compromised immune systems. The results may inform dietary recommendations for these vulnerable groups, ensuring that food safety measures do not come at the expense of essential nutrient intake. The study may also help in improving emergency food supply chains, particularly for disaster relief efforts where irradiated food products are used to ensure food safety and longevity.

Figure E1.2. *Difference in conditions for unirradiated and irradiated strawberries, kept at a temperature of 4°C for 15 days.*

Experiment Scenario

To assess the impact of gamma irradiation on the stability of ascorbic acid in local food products, a quantitative titration method will be employed.

This section may be skipped by the student if desired. The following steps have been carried out prior to this exam; it is provided with the intent of providing students with context regarding the experiment they are about to undertake.

<u>Preparation of solutions</u>

A. Metaphosphoric acid – acetic acid (HPO_3 – HAc)

i. Fill a 100-mL volumetric flask (VF) with 50 mL of distilled water.

ii. Dissolve 8 mL of HAc into the solution

iii. Dissolve 3 g of HPO_3 into the solution.

iv. Dilute the solution with distilled water up to the VF mark.

B. Dichlorophenol indophenol (DCPIP)

i. Fill a 1-L VF with 250 mL of distilled water.

ii. Dissolve 168 mg of sodium bicarbonate ($NaHCO_3$) into the solution.

iii. Dissolve 200 g of sodium DCPIP into the solution.

iv. Dilute the solution with distilled water up to the VF mark.

C. Standard ascorbic acid solution

i. Fill a 100-mL VF with 100 mL of HPO_3 – HAc solution

ii. Dissolve 25 mg of ascorbic acid into the solution.

iii. Repeat for different weights of ascorbic acid solution dissolved, up to 200 mg per 100 mL of HPO_3 – HAc solution.

iv. These solutions are required for plotting the standard curve.

D. Samples solutions

i. Dissolved ascorbic acid of known concentration (Standard solution).

ii. An unknown concentration of crystalline ascorbic acid, dissolved in distilled water after irradiation at various doses of 0, 1, 5, and 10 kGy.

Given Materials

Ensure that all the materials listed below are present and accounted for. Promptly inform the person in charge if any items are missing or incomplete, so that the necessary

arrangements can be made.

A. 1 unit of spray bottle of 2, 6 sodium dichlorophenol indophenol and sodium hydrogen carbonate solution (DCPIP) to act as the titrant.

B. Set A (Standard solution titration)

i. 12 tubes of 5-mL metaphosphoric and acetic acid solution (HPO_3 – HAc)
ii. 10 tubes of 2-mL ascorbic acid standard solution with different concentrations each. (25, 50, 100, 150, 200 mg/100 mL)
iii. 2 tubes of 2-mL blank solution (Distilled water)

C. Set B (Sample solution titration)

i. 8 tubes of 5-mL metaphosphoric and acetic acid solution (HPO_3 – HAc)
ii. 8 tubes of 2-mL ascorbic acid sample solution (unknown concentration) with different irradiation dose each (0, 1, 5, 10 kGy irradiation dose)

D. 2 units of conical flasks

E. 1 unit of burette

F. 1 unit of retort stand

G. 1 unit of spray bottle of distilled water

H. 1 unit of beaker for excessive DCPIP

I. 1 unit of beaker as waste container

Experiment Procedure

The following section provides a detailed guide on conducting the experiment. Students shall carefully follow each step within this section to ensure accurate results and a thorough understanding of the experimental process. Take the appropriate time to go through the materials and methods before proceeding.

E1.1 Titration of Solutions (3.8 pts)

A. Ascorbic acid standard solutions (Set A)

i. Fill in the burette with DCPIP and record the initial volume of DCPIP. The initial volume used is up to the participants.
ii. Pour in 5 mL of HPO_3 – HAc solution into a 100-cm^3 conical flask.
iii. Pour in 2 mL of blank solution into the flask.
iv. Titrate with DCPIP solution until a light-pink-coloured solution is produced and record the final amount of DCPIP. Repeat this step with another conical flask and record the relevant readings.
v. Discard the light pink solution into the waste container and rinse the conical flask using the wash bottle provided.
vi. Repeat for each concentration of standard ascorbic acid solution (25, 50, 100, 150 and 200 mg), replacing the blank solution.

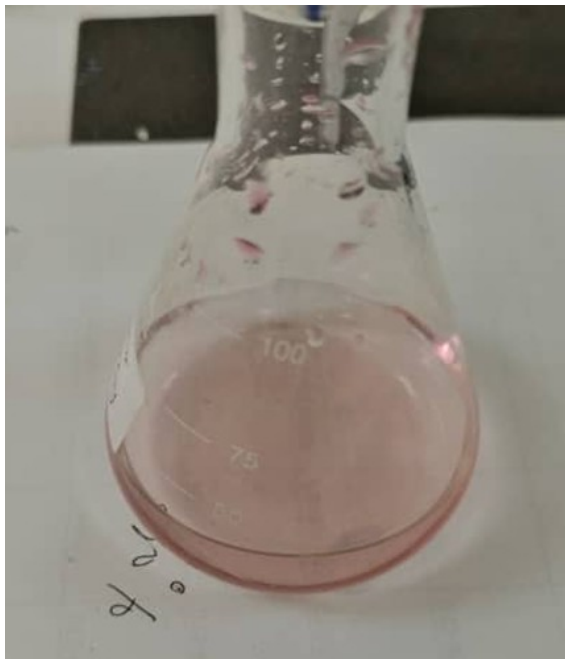

Figure E1.3. *The desired light-pink-colored solution after the titration process.*

E1.1.1	**Record** the amount of DCPIP solution used in each attempt for each standard solution of different concentrations.	1.8 pts

E1.1.2	Calculate the average volume of DCPIP used for each standard solution concentration. Include your calculations in the answer sheet.	0.3 pts

B. Ascorbic acid sample solutions (Set B)

i. Fill in the burette with DCPIP and **record** the initial volume of DCPIP. The initial volume used is up to the participants.
ii. Pour in 5 mL of HPO_3 – HAc solution into a 100-cm^3 conical flask.
iii. Pour in 2 mL of 0 kGy irradiated ascorbic acid solution into the flask.
iv. Titrate with DCPIP solution until a light-pink-coloured solution is produced and **record** the final amount of DCPIP. Repeat this step with another conical flask and **record** the relevant readings.
v. Discard the light pink solution into the waste container and rinse the conical flask using the wash bottle provided.
vi. Repeat for each sample ascorbic acid solutions (1, 5, 10 kGy).

E1.1.3	**Record** the volume of DCPIP solution used in each attempt for each sample solution.	1.2 pts

E1.1.4	Calculate the average volume of DCPIP used for each sample solution. Include your calculations in the answer sheet.	0.2 pts

E1.1.5	A student is performing a titration experiment to determine the concentration of ascorbic acid in an irradiated sample. However, their volume measurements show high variability across multiple trials, leading to inconsistent results. Which of the following is the most effective combination of methods to improve precision and reliability in this experiment? A. Increase the sample volume to reduce percentage error and record only the trial with the most consistent result. B. Use a burette with a higher precision scale and perform at least three trials to calculate the average. C. Conduct additional trials, but discard any results that do not closely match the expected concentration. D. Perform the titration in a single trial, ensuring that the endpoint color change is observed under bright lighting.	0.3 pts

E1.2 Determination of Ascorbic Acid Concentrations (3.6 pts)

With the data of standard and sample solutions collected from questions **E1.1.2** and **E1.1.4**, construct a curve to act as a reference for ascorbic acid concentration.

E1.2.1	Plot a graph of DCPIP solution volume used against the concentration of standard solution and draw the best fit line. Calculate the slope of the best fit line created. Graph paper is provided within the answer sheet.	1.0 pts
E.1.2.2	Using the graph from **E1.2.1**, determine the concentration of ascorbic acid present in the samples for each irradiation dose.	0.4 pts
E1.2.3	By referring to the initial unirradiated concentration of ascorbic acid as 100%, calculate the concentration percentage of each irradiated ascorbic acid sample. Concentration of control (unirradiated) ascorbic acid as 100%: $Concentration\ of\ sample\ ascorbic\ acid$ $= \frac{Average\ volume\ of\ DCPIP\ used}{Average\ volume\ of\ DCPIP\ used\ at\ 0\ kGy} \times 100\%$	0.2 pts
E1.2.4	Based on the calculations made in **E1.2.3** plot the stability curve of the sample concentration against irradiation doses, and calculate the slope of the best fit line created.	1.0 pts
E1.2.5	Using the stability curve from **E1.2.4**, determine the dose at which 50% of ascorbic acid in the sample ascorbic acid solution is degraded.	0.4 pts
E1.2.6	To evaluate the consistency of your titration measurements, determine the **mean volume** $(\overline{x})$and **standard deviation** (SD) of the DCPIP solution volume used for each sample. Use n-1 in the calculations for SD.	0.6 pts

E1.3 Stability of Ascorbic Acid under Irradiation (2.6 pts)

The degradation of ascorbic acid under gamma radiation is hypothesized to follow a combination of zero-order and first-order kinetics. The rate of degradation in relation to radiation dose can be modelled as:

$$R = k_0 + k_1 \times C$$

where k_0 is the zero-order rate constant, k_1 is the first-order rate constant, and C is the concentration of ascorbic acid. Assume the initial concentration of ascorbic acid is $0.025\ \mathrm{M}$, and initial experiments suggest $k_0 = 0.0015\ \mathrm{M/kGy}$ and $k_1 = 0.12\ \mathrm{kGy^{-1}}$.

The combined kinetics model for ascorbic acid degradation in relation to radiation dose is given by the differential equation:

$$\frac{dC}{dD} = -\left(k_0 + k_1 \times C\right)$$

E1.3.1	Derive an expression for C as a function of radiation dose D. Note that R is the rate of change of C with respect to D. **Show your working in the answer sheet.**	0.6 pts
E1.3.2	Calculate the concentration of ascorbic acid if $D = 15\ \mathrm{kGy}$.	0.2 pts
E1.3.3	If the permissible threshold for ascorbic acid concentration is 0.015 M, determine the maximum dose D that the solution can be subjected to before having its concentration reduced below the threshold? **Show your working in the answer sheet.**	0.4 pts

With the integrated solution for ascorbic acid degradation in relation to radiation dose:

$$C = \frac{k_0}{k_1}\left(e^{-k_1 D} - 1\right) + C_0 \times e^{-k_1 D}$$

E1.3.4	Find the initial rate of degradation (D=0), using the values from **E1.3**. (Information included in the beginning of section **E1.3**)	0.2 pts
E1.3.5	Derive the expression for $\frac{dC}{dD}$ as a function of dose D.	0.6 pts
E1.3.6	Set up an equation for dose D^* at which the degradation rate is halved, using the formula from **E1.3.5**.	0.2 pts
E1.3.7	Solve for D^* using the values from **E1.3**. (Information included in the beginning of section **E1.3**)	0.4 pts

E2 Locating and Handling a Missing Radioactive Source (10 pts)

A Cesium-137 radioactive source with an activity of 5 MBq has been misplaced within the university's nuclear instrumentation laboratory. This sealed source is typically used for instrument calibration and testing. Due to the potential health risk posed by gamma radiation, immediate action is required to locate and secure the source in accordance with radiation safety protocols.

The laboratory is equipped with four fixed area monitors (area monitors 1 to 4), which continuously record radiation count rates in count per second (CPS). A separate hallway monitor (area monitor 5) positioned outside the laboratory records background radiation levels. The laboratory layout and the coordinates (x_i, y_i) of each monitor are provided in Figure E2.1.

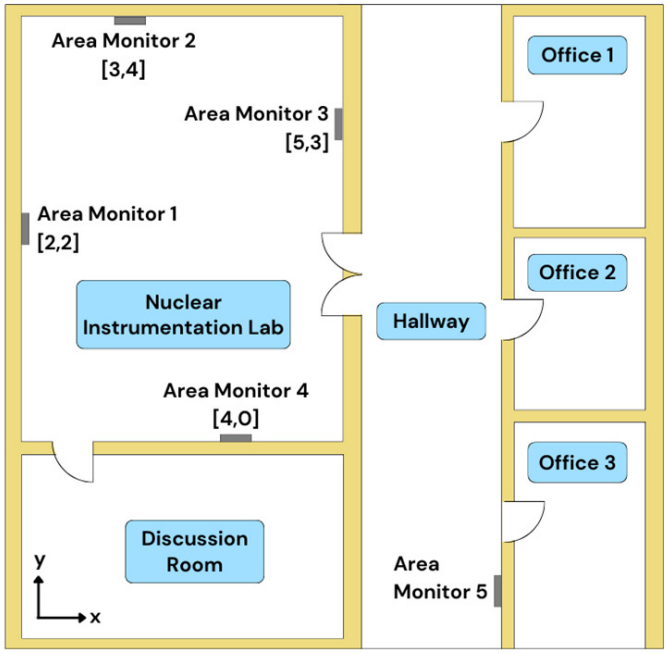

Figure E2.1. *Laboratory layout showing the positions and coordinates of the four fixed area monitors and the hallway background monitor.*

E2.1 Statistical Analysis of the Area Monitors Data (2.4 pts)

Radiation measurements are inherently variable due to the stochastic nature of radioactive decay. Although radiation data follows a Poisson distribution, when the average count rate is sufficiently high, the data can be approximated using a Gaussian distribution. This simplifies analysis while maintaining accuracy for higher count rates.

You are provided with 10 measurements for each of the four monitors. Area monitors 1, 2, 3, and 4 have an efficiency of 20% for detecting gamma rays emitted by Cesium-137. Meanwhile, the background radiation has been measured at an average of 13 CPS.

Table E2.1. *Recorded measurements of area monitors 1, 2, 3 and 4 in CPS.*

Area Monitor 1	Area Monitor 2	Area Monitor 3	Area Monitor 4
18025	42356	81987	8008
17008	36177	95035	10361
17755	39518	73222	8260
18147	42948	63689	8437
14340	46703	82352	10464
17452	41744	68199	7733
15756	33032	70211	8446
17481	47661	89085	10019
13186	44849	63728	8100
10130	23032	108392	8722

E2.1.1	Complete the table given in the answer sheet based on the provided data and information. Write the answers in two decimal places for standard deviation and relative uncertainty. Assume the underlying decay process follows Poisson distribution. Given; $Net\ Count\ Rate\ =\ Average\ Count\ Rate\ -\ Background\ Count\ Rate$ $True\ Count\ Rate = \frac{Net\ Count\ Rate}{Detector\ efficiency}$ $Standard\ deviation,\ \sigma = \sqrt{Net\ Count\ Rate}$ $Relative\ uncertainty = \frac{\sigma}{Net\ Count\ Rate} \times 100$	2.0 pts

E2.1.2	You observe new data of 20,000 CPS on area monitor 1 and 9,000 CPS on area monitor 4. To determine if the data are outliers requiring further investigation, use the following criteria of Z score: • If $Z > 2$, the data is considered an outlier and requires further investigation • If $Z \leq 2$, the data is considered a normal fluctuation and no immediate action is required Calculate the Z-score for each of the observed data and determine whether they should be considered a normal fluctuation or an outlier requiring investigation. Given; $Z\ score,\ Z = \frac{\chi - \mu}{\sigma}$	0.4 pts

E2.2 Identification of the Missing Source Location (2.6 pts)

The data from the area monitors suggests that the missing Cesium-137 source is still within the laboratory. By analyzing the radiation intensity data from the fixed area monitors, the distance from each monitor to the source can be estimated. Since the intensity of gamma radiation from a point source decreases with the square of the distance r from the source, the following equation is used:

$$I = \frac{A}{4\pi r^2}$$

Where:

I: Intensity at a distance *r* from source

A: Source Activity

r: Distance from the source to the detector

E2.2.1	Using the true count rate values, apply the inverse square law to calculate the distance of the source from each monitor (1 to 4). Write the answers in two significant figures.	0.6 pts
E2.2.2	Choose three area monitors with the smallest distance to the source. Next, plot and triangulate the position of the missing source (x_s, y_s) on a graph paper. You may assume the source is a point source.	1.0 pts
E2.2.3	Verify the missing source coordinates (x_s, y_s) by solving the simultaneous equations of the distance; $r^2 = (x_s - x_i)^2 + (y_s - y_i)^2$. Please show your steps.	1.0 pts

E2.3 Handling the Missing Source with ALARA Principle (2.3 pts)

When working with ionizing radiation, radiation exposure should be minimized to As Low As Reasonably Achievable (ALARA).This ALARA concept is applied to minimize the exposure and it is the fundamental principle in radiation protection that guides safe practices. There are three methods that are used for implementing the ALARA concept:

- Time: Minimize duration of exposure
- Distance: Maximize distance from radiation sources
- Shielding: Use appropriate materials to block radiation

You as a radiation worker must retrieve and secure the source while ensuring that the total exposure remains below 100 mR. The identified Cesium-137 source is emitting gamma radiation with an intensity of 500 mR/h at a distance of 1 meter from the source.

E2.3.1	If you plan to spend 30 minutes at a distance of 1 meter from the radiation source, calculate your total exposure in sieverts. Use the conversion factor: 1mR = 10 µSv	0.5 pts
E2.3.2	You are using a shielding material that reduces your exposure by 50%. Assuming that you are working at the same distance as in E2.3.1, calculate your total exposure after working for 30 minutes.	0.3 pts
E2.3.3	You need to maintain the same total exposure limit (100 mR) but are allowed to work closer to the source. If you move to a distance of 0.5 meters from the source, how much time can you work safely, considering the inverse square law of radiation?	1.0 pts
E2.3.4	As you are dealing with a sealed Cs-137 source, your primary concern is external exposure. However, if the source were unsealed, state three ways how Cs-137 could enter your body and lead to internal exposure.	0.3 pts
E2.3.5	A worker inhales Cs-137 particles with an activity of $1.0 \times 10^{6}\,\mathrm{Bq}$. It is assumed that the inhaled Cs-137 particles remain lodged in the respiratory tract for the entire year. Given that the dose conversion factor (DCF) for Cs-137 inhalation is $1.3 \times 10^{-8}\mathrm{Sv/Bq}$, calculate the worker's annual committed effective dose (CED) in Sieverts.	0.2 pts

E2.4 Creating Temporary Shielding for the Source (2.7 pts)

While retrieving the source, the temporary shielding should be constructed to minimize radiation exposure. A Geiger-Müller (GM) counter is used to measure the radiation count rate (in counts per minute, cpm) at different shielding thicknesses of lead. You are tasked to plot a graph according to Table E2.2 and determine the half-value layer, tenth-value layer, linear attenuation coefficient, and mass attenuation coefficient.

Table E2.2. *Measurement readings for different lead thicknesses.*

Shielding Thickness [cm]	First Reading [cpm]	Second Reading [cpm]
0	82,900	82,800
0.107	73,000	73,300
0.2	65,900	64,700
0.4	52,900	51,600
0.5	46,800	46,200
0.6	41,700	41,600
0.7	37,100	36,500
0.8	32,800	32,500

E2.4.1	Calculate average reading and net reading for each shielding thickness in Table E2.2. Given the background reading is 83.75 cpm. Please answer in two decimals places.	0.8 pts
E2.4.2	Plot a semi-log graph of net reading against shielding thickness.	0.5 pts
E2.4.3	Based on the graph, if the shielding thickness is 0.3 cm, determine the net reading in cpm.	0.1 pts
E2.4.4	From the graph, determine the half-value layer (HVL).	0.2 pts

E2.4.5	Calculate the linear attenuation coefficient of the lead shielding.	0.1 pts
E2.4.6	Determine the tenth-value layer of the lead shielding.	0.1 pts
E2.4.7	Determine the mass attenuation coefficient (μ_m) of the lead shielding. The density of lead is 11.35 g/cm^3	0.1 pts
E2.4.8	Calculate the required thickness in cm to reduce the count rate to 1% of its initial value. Please answer in one decimal place.	0.2 pts
E2.4.9	Calculate the mass (in kg) of the minimum thickness required for the lead shielding (from question at E2.4.8) to 1% shield the missing source around 1 m^2 area.	0.3 pts
E2.4.10	If a different material such as concrete (density = 2.33 g/cm^3, mass attenuation coefficient at 662 keV = 0.07 cm^2/g) were used instead of lead, determine the required thickness in cm of concrete to achieve count rate to 1% of its initial value. Please answer in one decimal place.	0.3 pts