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FRICKE DOSIMETRY IN LOW DOSE RANGE
FOR FOOD IRRADIATION

August 1982

Tamikazu KUME, Hiroyuki TACHIBANA
and Masaaki TAKEHISA

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Fricke Dosimetry in Low Dose Range for Food Irradiation

Tamikazu KUME, Hiroyuki TACHIBANA and Masaaki TAKEHISA

Food Irradiation Development Laboratory,
Takasaki Radiation Chemistry Research Establishment, JAERI

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In order to establish a suitable method of dosimetry in low dose range on food irradiation, the precision limit of the Fricke dosimeter (ferrous sulfate dosimeter) and the effect of water purification on Fricke dosimetry were investigated. At the dose range lower than 40 Gy, the errors of Fricke dosimetry are larger than 0.5 % but does not exceed 2 % at the dose as low as 15.5 Gy, whereas the errors are within 0.5 % at more than 57.6 Gy. Among various kinds of water (tap-water, single distilled water, Milli-Q water and triply distilled water), Milli-Q water could be use for the Fricke dosimeter as good as triply distilled water. Milli-Q water is easily prepared in a short time from a Milli-Q system with the use of ion-exchange cartridge and millipore filter. From these results, it has become apparent that Fricke dosimetry is applicable to low dose such as onion sprout inhibition (minimum dose 20 Gy).

Keywords: Fricke Dosimeter, Dosimetry, Low Dose Range, Food Irradiation, Precision Limit, Water Purification, Mutual Comparison, Irradiation Facility, Error

食品照射のための低線量域における Fricke 線量測定

日本原子力研究所高崎研究所食品照射開発試験室

久米 民和・橘 宏行・武久 正昭

(1982年7月19日受理)

食品照射領域における低線量の測定に適した線量測定法の確立を目的として、フリッケ線量計の精度限界および水の精製度の影響について検討した。57.6 Gy 以上の線量ではフリッケ線量計の誤差は0.5%以内であるのに対し、40 Gy 以下の線量範囲では誤差は0.5%より大きくなった。しかし、15.5 Gy といった低線量でも2%以内の誤差であった。したがって、フリッケ線量計を玉ネギの発芽防止（最低線量20 Gy）の測定に用いることは、十分可能であることが明らかとなった。また、精製度の異なる種々の水（水道水、Milli-Q 水、蒸留水、3回精製水）のうち、Milli-Q 水は3回精製水と同様フリッケ線量計に用いることが可能であった。Milli-Q 水はイオン交換カートリッジおよびミリポアフィルターを用いたシステムにより短時間で簡単に調製できる。これらの結果をもとに、低線量域の線量を精度よく簡便に測定するためのシステムを作成した。

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1. Introduction

For food irradiation dosimetry, the ferrous sulfate solution has been most frequently used and is recommended in IAEA Manual¹⁾ as a reference dosimeter. It has tissue-equivalent absorption characteristics, a linear dose versus effect relationship and good accuracy. It is, however, sensitive to impurities, and restricted for use in the dose range of 40 ~ 400 Gy (4 ~ 40 krad) with air saturation.²⁾

Since the dose range used for sprout inhibition is 20 ~ 100 Gy for onions³⁾ and 60 ~ 150 Gy for potatoes,⁴⁾ it is required to extend the low dose limit of the Fricke dosimeter. It has been pointed out^{2,5)} that the lower limit of the Fricke dosimeter can be extended with the same accuracy by using longer cells or by measuring the ferric absorbance at 224 nm where ferric ion absorbs more strongly than at 304 nm. But the use of a shorter measuring wavelength is undesirable because the scatter increases. Lower dose can also be measured by using radioactive ion (⁵⁹Fe) or by making a derivative of ferric ion that has a higher extinction than the ferric ion alone. However, these methods need somewhat tedious treatment.

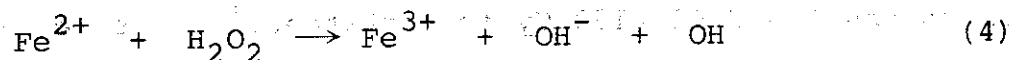
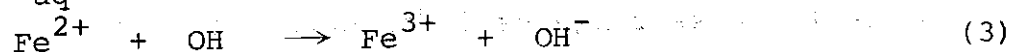
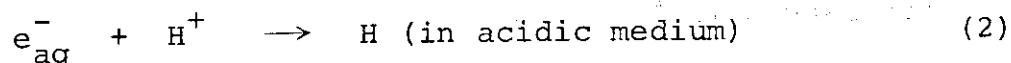
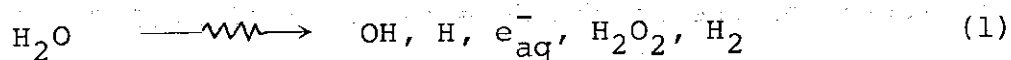
In this paper, we report the effect of water purification on Fricke dosimetry and the precision limit of the Fricke dosimeter in order to establish a suitable method for the measurement in low dose range for food irradiation. And this paper was prepared for the textbook of IAEA training course.

2. Principle²⁾

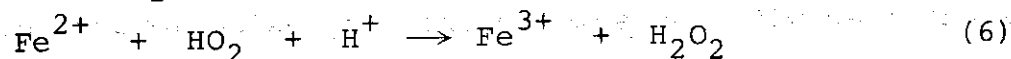
2.1 Radiation Chemistry of Ferrous Sulfate Solution

2.1.1 Oxidation of Ferrous Ion

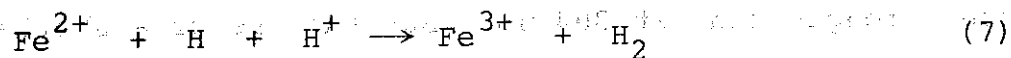
The reaction scheme of the oxidation of ferrous ion is usually assumed as follows:



a) in the presence of oxygen



b) in the absence of oxygen



On the basis of the above scheme, the total yield of ferric ion can be represented as follows:

a) in the presence of oxygen

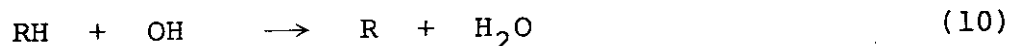
$$G(\text{Fe}^{3+}) = G(\text{OH}) + 2G(\text{H}_2\text{O}_2) + 3G(\text{H}) \quad (8)$$

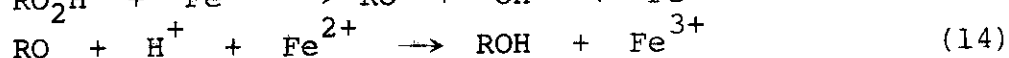
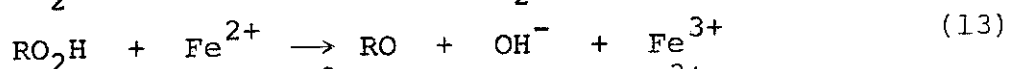
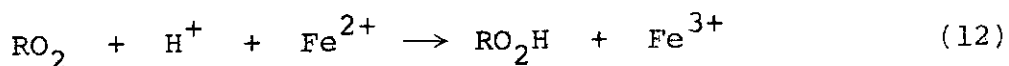
b) in the absence of oxygen

$$G(\text{Fe}^{3+}) = G(\text{OH}) + 2G(\text{H}_2\text{O}_2) + G(\text{H}) \quad (9)$$

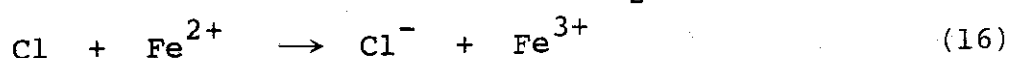
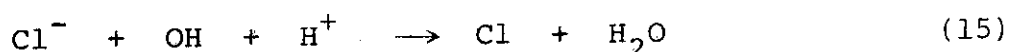
2.1.2 Effect of Sodium Chloride

When some organic impurities (RH) are present in the system, they usually react faster with OH radicals than with ferrous ions as follows:





The effect of organic impurities is to increase the yield of ion oxidized. In order to prevent the effect of organic impurities on the oxidation of ferrous ion, a small amount of sodium chloride is usually added in the system. When chlorine ions are present in the system, they react as follows:



The reactions (15) and (16) are equivalent to the reaction (3). Therefore, sodium chloride is usually added by 0.001 M as a stabilizer of the dosimeter.

2.2 Application of Fricke Dosimeter^{2,6)}

The application of the Fricke dosimeter are as follows:

- 1) The accuracy of the measurement of dose is not affected by dose rate up to $2 \times 10^6 \text{ Gy} \cdot \text{sec}^{-1}$ and of temperatures between 1 and 60°C during irradiation.
- 2) $G(\text{Fe}^{3+})$ is independent of sulfuric acid concentrations between 0.05 and 0.75 M and of ferrous ion concentrations between 10^{-4} and $5 \times 10^{-2} \text{ M}$.
- 3) The measurable dose is up to 0.4 kGy, 2 kGy and 20 kGy for the air-, oxygen-saturated and oxygen-free solutions, respectively.
- 4) The vessel for the Fricke dosimeter should have an inner diameter of at least 8 mm.

3. Experimental

3.1 Purification of Water

Since the yield of ferric ion is affected sensitively by organic impurities, triply distilled water is usually used. The water is firstly distilled from acidic permanganate solution, then from an alkaline permanganate solution and finally without the salt. The compositions of each solution are as follows;

1st; KMnO_4 1g, H_2SO_4 5ml/l

2nd; KMnO_4 0.1g, NaOH 5g/l

3rd; without the salt

To examine the effect of water purification, tap-water, single distilled water and Milli-Q water were used. Tap-water in the laboratory contains about 10 ppm Cl^- , 2 ppm organic impurities and small amount of metallic ions (Fe, Zn, etc.). Single distilled water was freshly prepared with Autostil WA-510 (Yamato Science Co. Ltd.) and Milli-Q water was obtained from a Milli-Q system (Millipore Co., USA) with the use of ion-exchange cartridge and millipore filter.

3.2 Preparation of Solution

The 0.4 M sulfuric acid solution containing 1×10^{-3} M ferrous ammonium sulfate and 1×10^{-3} M sodium chloride in triply distilled water was usually used. The solution containing high concentration of ferrous ammonium sulfate (4×10^{-2} M) was also used. Guaranteed-reagent grade chemicals were used without further purification.

$\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ (Mohr's salt) 0.195g (7.84g)

H_2SO_4 11.2ml

NaCl 0.030g /500ml

The vessels (test tubes, volumetric flasks, pipettes and bubbling bottles) were put into chromic acid mixture overnight and were washed with distilled water and finally triply distilled water.

3.3 Irradiation

Pyrex test tubes ($\phi 15 \times 150$ mm) were used for irradiation vessel. The tubes were irradiated about 10 kGy filling with ferrous sulfate solution before use. The ferrous sulfate solution was bubbled with oxygen for about 15 min before irradiation in order to saturate with oxygen. Gamma irradiation was mainly performed by means of 25 kCi ^{60}Co cylindrical source or 90 kCi slab source.

3.4 Analytical Method of Ferric Ion²⁾

The amount of ferric ion is determined by measuring the absorbance of the solution at 304 nm, at the peak of the absorption spectrum of ferric ion. That is, the ferric ion concentration ($[\text{Fe}^{3+}]$) is estimated by the equation (17).

$$[\text{Fe}^{3+}] \text{ (mol} \cdot \text{l}^{-1}\text{)} = \frac{\Delta A}{d \cdot \epsilon [1 + 0.007(T - 25)]} \quad (17)$$

where ΔA : Difference in absorbance between the irradiated and unirradiated solutions at 304 nm,

d : Optical path length (1cm),

ϵ : Molecular extinction coefficient at 304 nm and at standard temperature ($2196 \text{ l} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$ at 25°C),

T : Temperature of the absorbance measurement ($^\circ\text{C}$).

3.5 Estimation of Absorbed Dose^{1,2)}

The absorbed dose (D) can be estimated by equation (18):

$$D = \frac{\Delta A \cdot N}{d \cdot \epsilon [1 + 0.007(T - 25)] \cdot \rho \cdot G(\text{Fe}^{3+})} \times \frac{b}{k} \quad (18)$$

where N: Avogadro's number (6.023×10^{23} molecules·mol⁻¹),

ρ : Density of dosimeter solution (1.025 g·cm⁻³ for 0.4 M sulfuric acid solution),

$G(\text{Fe}^{3+})$: Radiation yield of ferric ion (15.5 for the aerated solution),

b: Energy conversion factor (1.602×10^{-14} rad·g·eV⁻¹),

k: Volume conversion factor (10³ cm³·l⁻¹).

Substituting the above values in the equation (18), the equation for the rapid calculation is represented in gray or rad unit as follows;

$$D(\text{Gy}) = \frac{2.77 \times 10^2 \Delta A}{1 + 0.007(T - 25)} \quad (\text{or } D(\text{rad}) = \frac{2.77 \times 10^4 \Delta A}{1 + 0.007(T - 25)}) \quad (19)$$

4. Results

4.1 Accuracy in the Low Dose Range

The ferrous sulfate solution was irradiated at the dose rate of 20.8 Gy/hr by using 25 kCi source. The relationship of the irradiation time and the change of absorbance at 304 nm was shown in Fig. 1. From this result, the following equation was obtained by the least-squares method.

$$\Delta A_{304} = 2.025 \times 10^{-1} t + 5.167 \times 10^{-3} \quad (20)$$

where t is the irradiation time in hr. Equation (20) has a small positive intercept, which is the dose during rising and falling times of the source. The same measurements were repeated six times

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where t is the irradiation time in hr. Equation (20) has a small positive intercept, which is the dose during rising and falling times of the source. The same measurements were repeated six times

and the data are shown in Table 1. The standard deviations for six data were $0.0020 \sim 0.0037$. The errors were calculated from the differences between the optical densities calculated from the equation (20) and experimental ones. At more than 57.6 Gy the errors were within 0.5 %.

The 4×10^{-2} M ferrous sulfate solution is used for high dose measurement. The accuracy at low dose range was studied using 4×10^{-2} M ferrous sulfate solution and the result are shown in Table 2. In this case, the absorbance at 304 nm for unirradiated solution was a little higher than that for 1×10^{-3} M ferrous sulfate solution but the difference of the results was not recognized.

4.2 Effect of Water Purification

In order to prevent the influence of organic impurities, the ferrous sulfate solution is usually prepared with triply distilled water and added a small amount of sodium chloride as stabilizer. However, the preparation of triply distilled water needs tedious treatment. The authors examined the effect of water purification and addition of sodium chloride. As shown in Table 3, the errors were within 1.5 % in the dose range of $43.2 \sim 179.0$ Gy for triply distilled water. On the other hand, the errors were larger with single distilled water or tap-water and the maximum error was 4 %. When the ferrous sulfate was dissolved in tap-water without sulfuric acid, the significant browning was observed. Tap-water contains impurities, but it is considered that the error is minimized by sodium chloride containing in the solution. Table 4 shows the

accuracy of the ferrous sulfate solution prepared with Milli-Q water or triply distilled water. In the case of sodium chloride addition, the errors were within 1.5 % for both water systems. In the absence of sodium chloride, the errors increased and the errors of triply distilled water was larger than that of Milli-Q water. The presence of impurities in triply distilled water was considered from this result. This would depend on the reason that the water was distilled immediately from alkaline permanganate solution without refluxing overnight.

4.3 Dose Measurement with Long Pathlength Cell

It has been pointed out that the lower limit of the Fricke dosimeter can be extended with the same accuracy by using longer cell. Table 5 compares the data measured with standard cell (1-cm) and long cell (5-cm). With use of 1-cm cell, the errors were 1.4 ~ 2.6 % in the dose range of 11.2 ~ 21.3 Gy, while those were lower than 0.8 % in the dose higher than 26.5 Gy. With use of 5-cm cell, the errors were always within 0.6 % in the dose range of 11.2 ~ 41.6 Gy. This result shows that higher precision could be obtained especially at the dose range below 20 Gy compared with 1-cm cell. However, it is possible to measure within the precision limit of 2 % at lower dose as 11 Gy with standard 1-cm cell.

4.4 Comparison with the Data at the Potato Irradiation Facility in Shihoro

The Fricke dosimeter is being used as a routine dosimeter at the Shihoro irradiation facility (Shihoro Agricultural Cooperation Association) where potatoes are irradiated commercially. Comparison of Fricke dosimetry data was made between Shihoro and JAERI. It took two weeks to transport the samples from JAERI to Shihoro and

return by air mail in February. As shown in Table 6, the errors caused by the difference of preparation place were 1.9 and 2.4 %. The values measured in Shihoro are little higher than those measured in JAERI, and overall maximum error is calculated to be 4.9 %. Taking into account the difference in the spectrophotometer used, measuring temperature, reagents, water and the effect of transportation, the results are in practically good agreement with each other. It has been reported that the effect of transportation for 42 days was not observed on Fricke dosimetry data.⁶⁾ These results demonstrate that the Fricke dosimeter can be used for the mutual comparison of the dosimetry among the irradiation facilities.

4.5 Application to the measurement of dose distribution in rice package

The dose distribution in package filled with rice was measured by Fricke dosimeter. The absorbed dose measured with two different type spectrophotometer are shown in Table 7. Since the irradiated dose was comparatively high in this experiment, the Fricke solution with high concentration of ferrous sulfate (4×10^{-2} M) was used and the optical density of irradiated solution was measured after five times dilution. Though the spectrophotometer, temperature and operator are different, the errors are within 2 % in twice experiments. However, the dilution technique is difficult and introduces an error especially for unskilled operator. When needed the dilution of the irradiated solution, skill in measurement is required.

5. Discussion

It has been described that the accuracy of the Fricke dosimeter is 1~2 % by Fricke and Hart,⁵⁾ 1.5 % in ICRU Report⁷⁾ and 1 % in IAEA Manual.¹⁾ In this experiment, relative error within 1.5 % was obtained at the dose more than 40 Gy which is reported lower limit in usual measurement. At the dose range lower than 40 Gy, the error becomes larger. This depends on the amount of oxidation products is not sufficient to be accurately measured. However, the error does not exceed 2 % at dose as low as 15.5 Gy (Table 1). It is therefore considered that the Fricke dosimeter would be applicable to the dosimetry for the low dose range of sprout inhibition without any modification. It is essential, however, that great care must be taken in the treatment especially when lower dose is to be measured. In order to obtain high precision, the purity of water is important. From the results in Table 5, Milli-Q water is found to be used for the Fricke dosimeter instead of triply distilled water. The Milli-Q system is expensive but it can prepare the purified water easily in a short time. In addition, appropriate precautions are needed for cleaning of the irradiation vessels, handling of absorption cells and measurement of temperature. Since a smudge across a cell window can easily introduce an error and the absorbance depends on the temperature, it would be desirable to use an autosampling cell with a constant temperature cell holder.

Using 5-cm cell, the accuracy within 0.6 % was obtained at lower dose of 11 Gy as shown in Table 5. Matthews et al⁸⁾ reported that the precision limit of within 2 % is attainable even for dose

as low as 1.2 Gy with use of 5-cm cell. However, the measurement using long cell is inconvenient for routine dosimetry on food irradiation because it needs a large amount of the Fricke solution. So the long cell could be used only when more severe accuracy is required.

In conclusion, it becomes apparent that the errors of Fricke dosimetry is within 2 % even at low dose as 15.5 Gy. Therefore, the Fricke dosimeter is applicable to the dosimetry for sprout inhibition without any modification. At present, the following Fricke dosimetry system is adopted in our laboratory to minimize the error. The Fricke solution is prepared with Milli-Q water and the optical densities are measured in a thermostated room by using a digital recording spectrophotometer and a flow-cell. With use of this system, the routine dosimetry for food irradiation can be carried out conveniently with high accuracy.

Acknowledgment

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- 2) J. W. T. Spinks and R. J. Woods: "An Introduction to Radiation Chemistry", John Wiley and Sons, Inc., New York, p.91 (1976).

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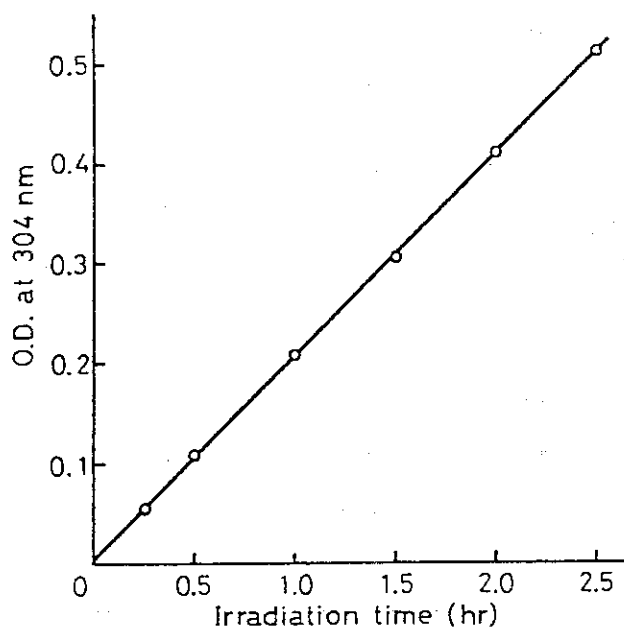


Fig. 1 Relationship between absorbance at 304 nm and irradiation time

Table 1 Relative errors for the Fricke dosimeter at low doses

Irradiation time (hr)	ΔA_{304} (exp.)	ΔA_{304}^{**} (calc.)	Dose ^{**} (Gy)	Error (%)
0.25	$0.055 \pm 0.0023^*$	0.056	15.5	1.8
0.5	0.108 ± 0.0027	0.106	29.4	1.9
1.0	0.207 ± 0.0020	0.208	57.6	0.5
1.5	0.308 ± 0.0027	0.309	85.6	0.3
2.0	0.411 ± 0.0037	0.410	113.6	0.2
2.5	0.512 ± 0.0029	0.511	141.5	0.2

Optical density at 304 nm was measured at 25.0°C.

* Standard deviation

** Calculated value by equation (20).

Table 2 Relative errors for the Fricke dosimeter with
 4×10^{-2} M ferrous sulfate

Irradiation time (hr)	Dose (Gy)	ΔA_{304} (calc.)	ΔA_{304} (exp.)	Error (%)
0.5	31.3	0.113	0.115	1.8
1.0	59.2	0.214	0.212	0.9
1.5	87.3	0.315	0.313	0.6
2.0	115.0	0.415	0.414	0.2
2.5	142.9	0.516	0.515	0.2

Absorbance at 304 nm was measured at 25°C.

Table 3 Effect of water quality on Fricke dosimetry

Irradiation time (min)	Dose (Gy)	ΔA_{304} (calc.)	Triply distilled water		Single distilled water		Tap water	
			ΔA_{304}	Error (%)	ΔA_{304}	Error (%)	ΔA_{304}	Error (%)
2	43.2	0.151	0.149	1.3	0.148	2.0	0.145	4.0
3	60.1	0.210	0.213	1.4	0.218	3.8	0.206	1.9
4	76.9	0.269	0.270	0.4	0.274	1.6	0.266	1.1
5	94.1	0.329	0.331	0.6	0.334	1.6	0.323	1.8
6	111.0	0.388	0.390	0.5	0.395	1.8	0.380	2.1
7	128.1	0.448	0.449	0.2	0.457	2.0	0.441	1.6
8	145.0	0.507	0.505	0.4	0.511	0.8	0.496	2.2
9	161.9	0.566	0.568	0.4	0.574	1.4	0.561	0.9
10	179.0	0.626	0.627	0.2	0.630	0.6	0.613	2.1

Absorbance at 304 nm was measured at 20.5°C.

Table 4 Effect of triply distilled water and Milli-Q water on Fricke dosimetry

Irradiation time (min)	Dose (Gy)	ΔA_{304} (calc.)	Triply distilled water				Milli-Q water			
			with NaCl		without NaCl		with NaCl		Without NaCl	
			ΔA_{304}	Error (%)	ΔA_{304}	Error (%)	ΔA_{304}	Error (%)	ΔA_{304}	Error (%)
2	43.2	0.151	0.149	1.3	0.161	6.6	0.149	1.3	0.157	4.0
3	60.1	0.210	0.213	1.4	0.220	11.6	0.208	1.0	0.213	1.4
4	76.9	0.269	0.270	0.4	0.283	5.2	0.270	0.4	0.274	1.6
5	94.1	0.329	0.331	0.6	0.344	4.6	0.329	0	0.334	1.6
6	111.0	0.388	0.390	0.5	0.406	4.6	0.388	0	0.393	1.3
7	128.1	0.448	0.449	0.2	0.467	4.2	0.446	0.4	0.454	1.3
8	145.0	0.507	0.505	0.4	0.531	4.7	0.505	0.4	0.516	1.8
9	161.9	0.566	0.568	0.4	0.587	3.7	0.565	0.2	0.574	1.4
10	179.0	0.626	0.627	0.2	0.648	3.5	0.624	0.3	0.638	1.9

Absorbance at 304 nm was measured at 20.5°C.

Table 5 Effect of long pathlength cell for measurement of optical density at low doses

Irradiation time (min)	Dose (Gy)	5-cm cell			1-cm cell		
		ΔA_{304} (calc.)	ΔA_{304} (exp.)	Error (%)	ΔA_{304} (calc.)	ΔA_{304} (exp.)	Error (%)
4	11.2	0.194	0.193	0.5	0.039	0.038	2.6
6	16.1	0.282	0.283	0.4	0.056	0.057	1.8
8	21.3	0.370	0.372	0.5	0.074	0.075	1.4
10	26.5	0.458	0.457	0.2	0.092	0.092	0
12	31.4	0.546	0.549	0.5	0.109	0.109	0
14	36.6	0.634	0.635	0.2	0.127	0.128	0.8
16	41.5	0.722	0.718	0.6	0.144	0.145	0.7

Absorbance at 304 nm was measured at 19.6°C.

Table 6 Comparison of Fricke dosimetry data between irradiation facility in Shihoro and JAERI

Preparation	Measurement	Temperature (°C)	ΔA_{304}	Dose (Gy)	Error (%)
JAERI	Shihoro*	22.0	0.521	147.4	1.9
Shihoro	Shihoro*	22.0	0.531	150.2	
JAERI	JAERI	20.3	0.498	142.6	2.5
Shihoro	JAERI	20.3	0.510	146.1	

Irradiation was carried out at the Shihoro irradiation facility using a 300 kCi ^{60}Co source.

* Measurements of the absorbance in Shihoro were made with a Hitachi 124 spectrophotometer.

Table 7 Dose distribution measurement in rice package with two different type spectrophotometer

	Position in package	Type 139*		Type 239**		Error (%)
		ΔA_{304}	Dose (Gy)	ΔA_{304}	Dose (Gy)	
Exp.1	A(front)	0.384	533	0.376	538	0.9
	B(middle)	0.206	287	0.204	292	1.7
	C(back)	0.103	145	0.101	144	0.7
	Temp.	25.0°C		20.6°C		
Exp.2	A(front)	0.387	540	0.388	549	1.7
	B(middle)	0.191	266	0.191	269	1.1
	C(back)	0.085	119	0.083	117	1.7
	Temp.	24.2°C		22.0°C		

* Normal manual type Hitachi 139 spectrophotometer,

** Digital type Hitachi 239 spectrophotometer with autosampling cell.