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Intercomparison of Luminescence Measurements of Bricks from Dolon' Village: Experimental Methodology and Results of European Study Group

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Luminescence/Retrospective dosimetry/Dolon'/Semipalatinsk/Fallout.

Thermoluminescence (TL) and optically stimulated luminescence (OSL) techniques were applied to quartz grains extracted from various depths in bricks taken from buildings in the village of Dolon', Kazakhstan, to determine the cumulative absorbed dose, D_T. The measurements were performed in four laboratories (EU supported Measurement Group). The results obtained using TL and OSL are compared and discussed with reference to known sources of experimental uncertainty and relevant luminescence characteristics that may affect the evaluation of the absorbed dose. The external nature of the irradiation due to gamma rays from artificial radionuclides is verified by the measurement of depth-dose profiles, and these are compared with those obtained in previous studies for bricks from the same region. To produce these profiles, the cumulative dose due to natural background radiation, D_{BG}, was assessed, particularly based on the concentrations of radionuclides of lithogenic origin within the bricks and the surrounding environment. The consistency of these estimates of D_{BG} was assessed using depth-dose data and absorbed dose determinations for bricks collected from shielded locations. The values of cumulative absorbed dose since the onset of fallout, D_X, were calculated as the difference between the values of total cumulative dose in bricks since its manufacture, D_T, and values of D_{BG}. Furthermore, estimations of the cumulative absorbed doses in air in the reference location near the sampled buildings, ${}_{RL}D_X$, were obtained using corresponding conversion factors, C_{RL}, estimated on the basis of previous work. In addition to results for samples distributed for the International Intercomparison, reference is made to relevant results from samples that were collected in 1999 from Dolon' village and studied as part of EU-supported research.

INTRODUCTION

Dolon' is one of the populated settlements downwind of the Semipalatinsk Nuclear Test (SNTS) site that has acquired one of the highest levels of fallout[1,2] arising from the nuclear test programme, in particular from the 1949 test. It has been the focus of interest for many studies concerning health effects as well as for dose reconstruction problems[3–6] including the application of the luminescence method with bricks from various locations.[7–9] The potential of the luminescence method applied to the areas affected by fallout from the SNTS was demonstrated in recent work by Bailiff *et al* 2004,[9] and it is relevant to note here that they commented on differences between some of the results obtained by different laboratories that exceeded the estimated limits of experimental error. In the present work a set of four brick samples from Dolon' was received by (or distributed to) the following laboratories (comprising the EU supported Measurement Group) as part of an International Intercomparison:

1. GSF-National Research Center for Environment and Health, Institute of Radiation Protection, Germany.
2. MRRC - Medical Radiological Research Center of RAMS, Obninsk, Russia.
3. Helsinki - Dating Laboratory, University of Helsinki, Finland (UHEL).
4. Durham - Luminescence Laboratory, Environmental Research Centre, University of Durham, United Kingdom (UDUR).

Each laboratory received a cut section of each brick that had been collected from different locations in Dolon' and per-

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formed luminescence measurements using their preferred techniques. For the work discussed in this paper, the luminescence measurements were made with quartz extracted from brick slices cut at specific depths from the exposed surface and of controlled thickness. In addition, the cumulative background dose due to natural sources of radiation was assessed using procedures that have been described previously[9] and also by applying a new approach (GSF laboratory) that is based on an analysis of the depth dose profile. The dose due to fallout was calculated and compared with the previously published results[9] for Dolon'.

MATERIALS AND METODS

The cumulative absorbed dose in brick due to fallout (D_X) is the difference between the cumulative dose measured using luminescence (D_T) and the dose due to natural sources of radiation D_{BG}. D_{BG} is generally calculated[10] using the following equation:

$$D_{BG} = A(a\dot{D}_\alpha + b\dot{D}_\beta + g\dot{D}_\gamma + \dot{D}_c) \quad (1)$$

where A is the age of the sample in years, a $\dot{D}\alpha$, b $\dot{D}_\beta$, and $\dot{D}g_\gamma$ are the effective dose-rate contributions (mGy/y) due to natural radionuclides arising from alpha and beta particles and photons, respectively, and D_c is the dose contribution due to cosmic radiation at a given latitude and altitude.[11] For the measurements discussed in this paper the alpha component of the natural dose had been removed by etching quartz grains (> 100 µm diameter) in hydrofluoric acid (HF). Further details of the reconstruction methodology can be found in numerous publications.[9,10,12,13] Since the age of the samples issued for the intercomparison were not estimated on the base of archive records, D_{BG} was assessed using an iterative procedure based on a comparison of depth-dose profiles obtained from Monte Carlo (MC) simulations of photon transport[14] with those obtained experimentally using luminescence.

Samples

Each laboratory received a slice from each of four bricks (size approximately 3 cm × 7 cm × 12 cm deep) from Dolon' for luminescence measurements. One of the samples (KSD 2-1, Large Church) was distributed in 2002, and the other three samples, KSD 1-3, Large Church, KSD 3-2, Small Church and KSD 4-1, School were distributed in 2003.[15]

Luminescence measurements

Measurements were performed with luminescence readers of similar type;[16,17] they contain a beta radiation source with which known absorbed doses can be administered to measurement aliquots. The beta sources were calibrated against a common secondary standard ^{60}Co photon source at GSF using quartz grains of various grain sizes.[18] Further technical details of the measurement conditions are summarised in Table 1.

Thermoluminescence measurements

Two laboratories (GSF and MRRC) measured the thermoluminescence (TL) signals and applied the Additive Multiple Aliquot Regenerative (ADMAR) and Multiple Aliquot Regenerative (MAR) dose procedures, using 6 and 8 aliquots respectively (~10 mg each). The measurement conditions, such as the optimum pre-heat and maximum heating temperatures were selected in preliminary tests before applying the MAR procedure.[12] The TL signals detected were brighter than typical samples, and the luminescence

Table 1. The luminescence techniques and measurement parameters used by the laboratories.

Laboratory	Method	Tech.	Grain size (µm)	Beta source Dose-rate (mGy/s)	Pre-heat (°C) Heating rate (°C/s)	Detection Filter	Tmax (°C)
MRRC	TL	MAR	150–250	9.12	160°C 2°C/s	Schott BG-39	350
GSF	TL	ADMAR	140–200	4.43	190°C 2°C/s	Schott BG-12 2mm	350
UDUR	OSL Blue/green	SAR	90–150	61.5	220°C hold 10s 240°C hold 10s	U-340	–
UHEL	Blue OSL at 120°C	SAR	210–300	31.0	280°C hold 10 s Test Dose PH 160°C	U-340 6mm	–

Table 2. Values of D_T obtained by the laboratories indicated for brick slices at the depths indicated, sample KSD-2- 1 (Large Church).

Depth (mm)	GSF Dose (mGy) OSL	UDUR Dose (mGy) TL	MRRC Dose (mGy) TL	UHEL Dose (mGy) OSL
1 ± 2	573 ± 39			
3.5 ± 1.5			596 ± 95	
5 ± 2	557 ± 40			
10 ± 2	522 ± 38	602 ± 18	518 ± 36	520 ± 30
20 ± 2	463 ± 30			
40 ± 2	445 ± 30			
60 ± 2	427 ± 30		475 ± 28	420 ± 28
80 ± 2	400 ± 30		480 ± 32	
100 ± 2	387 ± 27	468 ± 39	362 ± 62	
120 ± 2			342 ± 41	350 ± 30

characteristics were found to be generally uniform except for one sub-surface sample measured by one laboratory (MRRC) at 3.5 mm for sample KSD 2-1. The values of the total dose at specified depths, D_T, are given in Table 2–5.

OSL measurements

OSL measurements (Durham and Helsinki) were performed using a Single Aliquot Regeneration (SAR) procedure[12,19] that, in the case of the Durham measurements, also included a correction for thermal transfer effects during pre-heating. Generally, 9-12 aliquots were measured and strong OSL signals were recorded. Durham's procedure included two pre-heat temperatures (220 and 240°C, hold 10s) and a range of administered doses that were 80–120% of the value of D_T estimated in preliminary tests. The average of the D_T values obtained for the two preheat treatments, was used in further calculations, on the assumption that there was no systematic variation in D_T with preheat temperature.

Table 3. Values of D_T obtained by the laboratories indicated for brick slices at the depths indicated, sample KSD-1-3 (Large Church).

Depth (mm)	GSF Dose (mGy) TL	UDUR Dose (mGy) OSL	MRRC Dose (mGy) TL
2 ± 2	558 ± 33		
3.5 ± 1.5			547 ± 36
5 ± 2	549 ± 32		
10 ± 2	542 ± 32	675 ± 32	537 ± 35
20 ± 2	533 ± 31		
40 ± 2	497 ± 29		
55 ± 5			459 ± 39
60 ± 2	464 ± 27		
80 ± 2	455 ± 27		
100 ± 2	429 ± 25	532 ± 43	
120 ± 2	416 ± 26		430 ± 39

Table 4. Values of D_T obtained by the laboratories indicated for brick slices at the depths indicated, sample KSD-3-2 (Small Church)

Depth (mm)	GSF Dose (mGy) TL	UDUR Dose (mGy) OSL	UHEL Dose (mGy) OSL
2 ± 2	598 ± 36		
5 ± 2	583 ± 35		
10 ± 2	552 ± 33	693 ± 30	560 ± 40
20 ± 2	516 ± 30		
40 ± 2	484 ± 29		
60 ± 2	460 ± 27		
65 ± 10			500 ± 30
80 ± 2	451 ± 27		
100 ± 2	440 ± 26	551 ± 25	
125 ± 10			440 ± 30

Table 5. Values of D_T obtained by the laboratories indicated for brick slices at the depths indicated, sample KSD-4-1 (The School)

Depth (mm)	GSF Dose (mGy) TL	UDUR Dose (mGy) OSL	MRRC Dose (mGy) TL	UHEL Dose (mGy) OSL
2 ± 2	553 ± 33			
3.5 ± 1.5			570 ± 51	
5 ± 2	583 ± 34			
10 ± 2	552 ± 30	598 ± 39	505 ± 43	470 ± 55
20 ± 2	516 ± 29			
40 ± 2	484 ± 21			
60 ± 2	460 ± 25		352 ± 45	380 ± 60
80 ± 2	451 ± 26			
100 ± 2	440 ± 26	445 ± 31		
120 ± 5			304 ± 39	330 ± 60

RESULTS AND DISCUSSION

Cumulative dose at various depths in bricks

The values of the total dose, D_T, determined for a specified depth range, are listed in Tables 2–5 and shown graphically in Figs 2a–d. The uncertainty associated with the mean values of D_T, given at the 1σ level of confidence, is less than 10%, except for one sample noted above (KSD 2 - 1, depth 3.5 mm) where it exceeded 15%. The precision in D_T given by each laboratory at a sample depth of 10 mm ranges between 6 and 10% and this is equivalent to the variance between the laboratories, and the mean values of D_T determined by all laboratories at this depth are summarised in Table 6, where the inter and intra variance of the mean values are indicated.

It is to be noted that the values of D_T obtained for KSD-

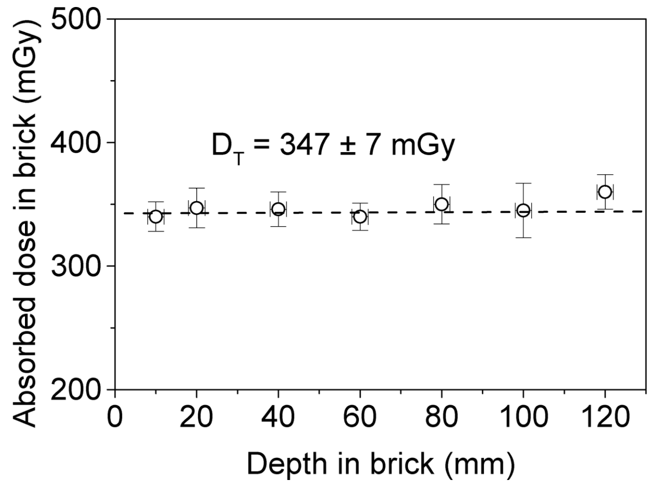

Fig. 1. Depth-dose profile in a brick collected from an inner wall of the former Large Church.

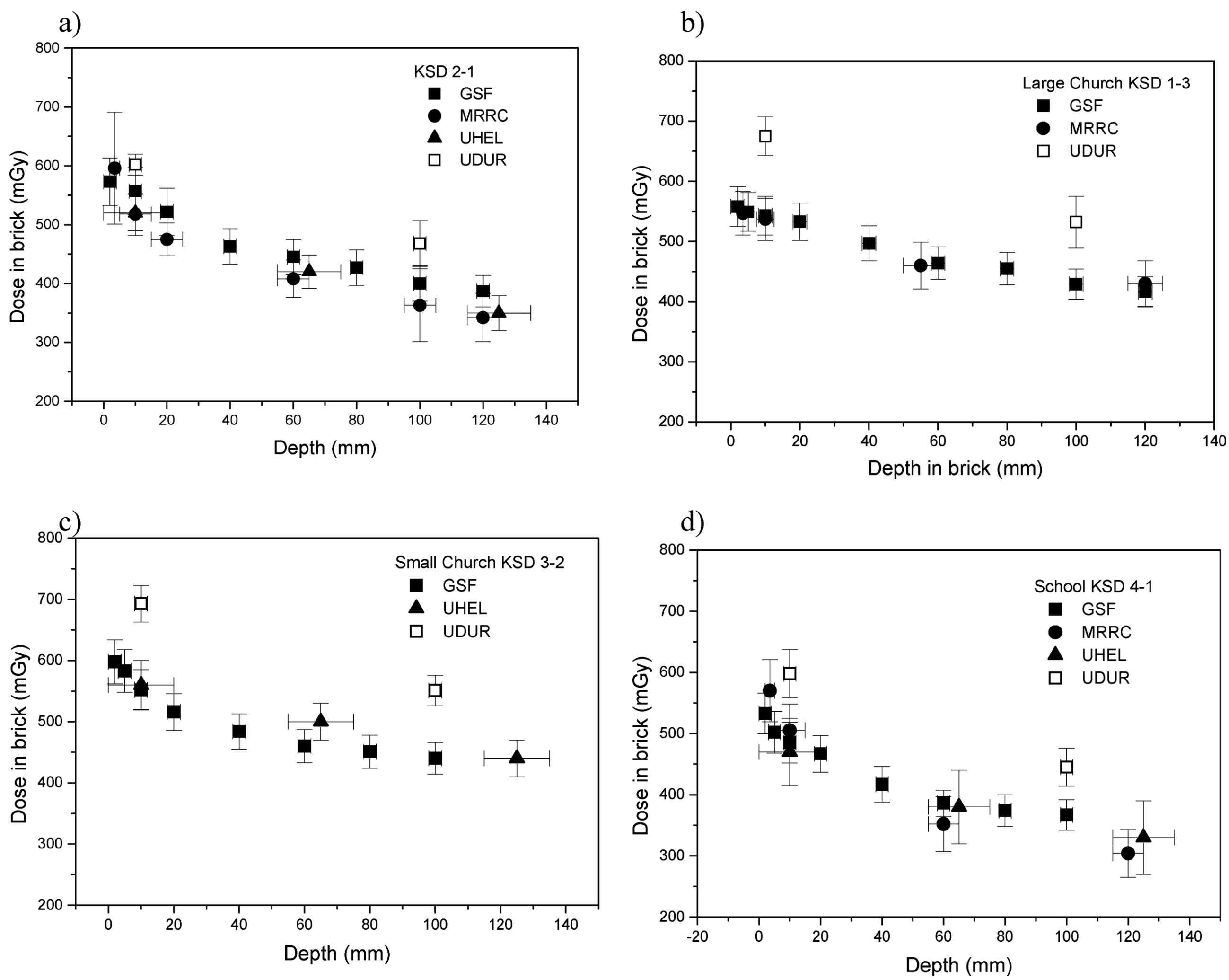

Fig. 2. Graphical representation of the depth-dose distribution measured using TL and OSL for various sections of a brick from a) the Large Church, sample KSD-2-1; b) the Large Church, sample KSD-1-3; c) the Small Church, sample KSD-3-2 and d) the School, sample KSD-4-1.

1 and KSD-2 by one laboratory (DUR) were systematically higher (by 25%) than the measurements performed by other laboratories. However, supplementary tests performed with quartz grains that had been photon irradiated at the GSF Secondary Standards Dosimetry laboratory confirmed that this difference was not connected with laboratory source calibration. Although the cause of this difference remains to be investigated, the unweighted mean of the D_T values obtained by all the laboratories for these two locations were within 1σ (68% level of confidence), whereas for the Large Church and the Small Church they were within 2σ limits.

Table 6. Values of the fallout dose, D_X, in brick at a depth of 10 mm from the exposed surface obtained by calculating the difference (D_T-D_{BG}) for the same depth, as discussed in the main text. In the first column the first error is the variance between laboratories and the second given in parenthesis is the variance in the mean value. $_{RL}D_X$ represents the cumulative dose in the air at the reference location.

Sample	D_T at 10 mm (mGy)	D_{BG} (mGy)	D_X (mGy)	$_{RL}D_X$ (mGy)
KSD-2-1 Large Church	540 ± 35(30)	350 ± 10	190 ± 36	494 ± 96
KSD-1-3 Large Church	584 ± 63(33)	390 ± 10	194 ± 65	504 ± 169
KSD-3-2 Small Church	602 ± 64(50)	380 ± 10	222 ± 65	577 ± 169
KSD-4-1 School	531 ± 48 (42)	320 ± 10	210 ± 50	546 ± 100

Assessment of cumulative natural background dose

In the absence of archive information concerning the ages for the sampled buildings, D_{BG} for each brick was estimated using an alternative procedure. The value of D_{BG} for each brick was obtained using a form of iterative analysis where the value of D_{BG} was varied until the calculated (based on Monte Carlo simulations[14]) and experimental (D_T-D_{BG}) depth-dose profiles matched – as illustrated in Figs 2a–d. To avoid potential systematic differences between laboratories that might affect the form of the depth-dose profile, only the results of one laboratory (GSF) were used for this analysis and the results are listed in Table 7. It should be noted that the iterative analysis used in these samples is based on the following assumptions:

a) The artificial radionuclide sources contributing to the fallout dose were distributed in the ground (1–30 g cm^{-2}), with an average photon energy similar to that of ^{137}Cs (i.e. 662 keV). According to previous work,[20] the time-averaged source energy from 1949 tests ranged between 500 and 800 keV.
b) D_{BG} does not vary with depth in the brick (10–120 mm).

Table 7. Summary of data related to the assessment of D_{BG} based on the concentrations of lithogenic radionuclides (cols 2-4) in bricks, measured using the techniques of thick source alpha counting and beta TLD. The beta, gamma and combined dose-rates at a depth of 10 mm in the wall calculated using these radionuclide concentrations is given in cols 5-7. The cumulative natural background dose derived from an analysis of the depth-dose profile is given in col. 9.

Sample	U (ppm) brick	Th (ppm) (mGy /y) brick	K_2O (%) (mGy /y) brick	Dose-rate $\dot{D}_{\gamma+c}$ (mGy)	Dose-rate $\dot{D}_\beta$ (mGy/y)	Assessed age (years)	D_{BG} (mGy)	D'_{BG} (mGy)
KSD-2-1 Large Church (2002)	6.92	3.43	2.49	1.26 ± 0.4	2.38 ± 0.04	96 ± 6	350	350 ± 10
KSD-1-3 Large Church (2003)	8.07	3.54	2.66	1.13 ± 0.3	2.55 ± 0.04	105 ± 7	386	390 ± 10
KSD-3-2 Small Church (2003)	4.68	1.52	3.13	1.16 ± 0.4	2.51 ± 0.06	103 ± 6	378	380 ± 10
KSD-4-1 School (2003)	6.18	3.88	2.64	1.19 ± 0.4	2.61 ± 0.07	84 ± 5	320	320 ± 10

* D_{BG}, the dose due to natural sources of radiation, was calculated using the conventional approach,[9] and corresponds to product of the sum of contributions from beta (col. 6), gamma and cosmic (col. 5) radiation from sources in the brick wall, soil and environment and the assessed age of the building. The radionuclide content of the soil adjacent to the building was assumed to be typical for the region and the thickness of the wall was assumed to be 25 cm.
** D'_{BG} was estimated using an iterative procedure, as discussed in the main text.

On the basis of a depth-dose profile (Fig. 1) for a brick from a shielded location in the former church in Dolon', this appears to be a reasonable approximation.

c) The brick samples were taken at a height of 1m above ground level.

Following the conventional approach, the average concentrations of (natural) lithogenic radionuclides in each brick were also determined using thick source alpha counting (TSAC) and β-TLD,[21,22] and the dose-rate was calculated for a depth of 10 mm from the brick surface (Table 7) using published conversion coefficients.[23] For each location, the age (Table 7) was estimated by calculating the quotient of D_{BG} derived from the profile data and the (natural) dose-rate. It can be seen that the ages estimated using this procedure are similar, within the uncertainties given, for samples from locations KSD 2-1, 1-3 and 3-2, whereas that from the school (KSD 4-1) is slightly younger.

Fallout dose in brick and conversion of dose in brick to dose in air

The values of D_X for a depth of 10 mm are shown in Table 6. It was assumed that the value of D_{BG} obtained for each brick using the iterative profile procedure described above applied to the sections of brick tested by each laboratory. It

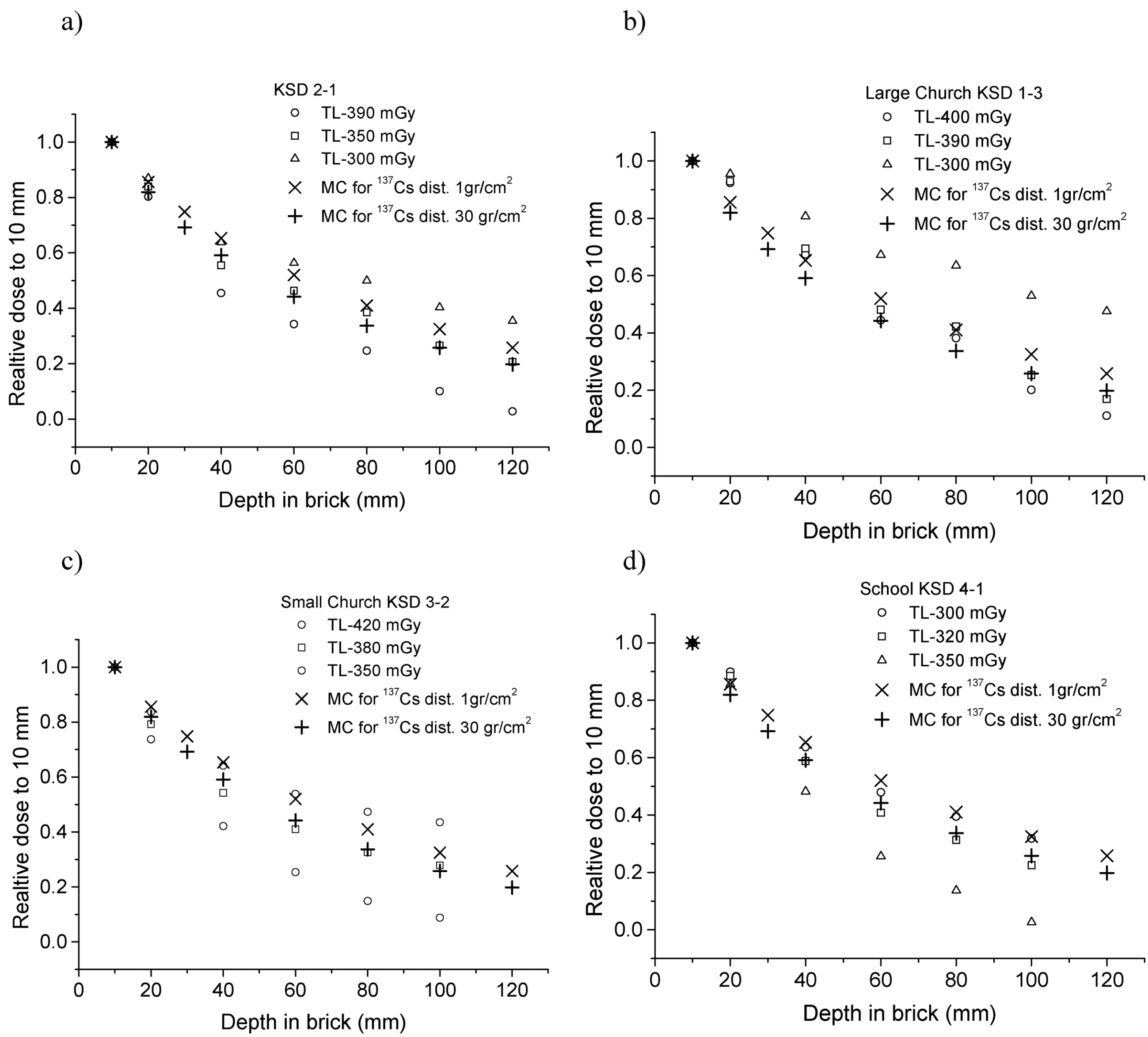

Fig. 3. Relative depth-dose distribution obtained using MC simulations and D_T-D_{BG} curves plotted for incremental variation of D_{BG} values. The best fits were obtained a) at 350 mGy for sample KSD-2-1, the Large Church, b) at 390 mGy for sample KSD-1-3, the Large Church, c) at 380 mGy sample KSD- 3-2, the Small Church, and d) at 320 mGy for sample KSD-4-1, the School.

is interesting to note that the average value of D_X for all four bricks (10–20 mm depth in brick) is 200 mGy, and this is similar to previously published values of D_X obtained by our group (5–15 mm depth in brick) for one building tested in Dolon'.

The overall error associated with D_x (68% level of confidence), was calculated by taking into account both random measurement errors and estimated systematic errors.[9] The values of D_T obtained for the sub-surface brick layers (e.g. 10 ± 2 mm) are the most appropriate to be used in this calculation since there is an unavoidable increase in the overall uncertainty associated with D_X where D_{BG} is a high proportion of D_T, which occurs at greater depths in the brick.[10] Since there is a measurable difference between D_T and D_{BG}, the value of D_X can be converted to absorbed dose in air at the Reference Location, ${}_{RL}D_X$, where,

$$ {}_{RL}D_x = C_{RL} \cdot D_x \qquad (2) $$

In contrast to work in the Chernobyl region,[13] the concentrations of extant artificial radionuclides in soils adjacent to the sampled buildings (and elsewhere) are not sufficient to estimate the original activity of deposition or, moreover, to assess localised heterogeneity in the distribution of fallout. Based on Monte Carlo simulations radionuclides for E = 662 keV deposited within about 20 m of the sample wall[9] account for about 60% of the absorbed dose in brick, the latter being most strongly influenced by variation in the concentration of radionuclides within this area. For the locations of relevance to the inter-comparison study, uniformity of deposition is assumed in the absence of extant data on fallout radionuclides, and consequently the coefficient F_h that was introduced to correct for such localised variation[13] cannot be applied. However, the sensitivity of the absorbed dose in brick to specified variation in radionuclide concentration can be explored by means of Monte Carlo simulations.

The conversion factor C_{RL} is obtained for a specified mean photon energy and source geometry using Monte Carlo simulations. For brick sampled in the middle of a plane wall at a height of 1m above ground level and with artificial radionuclide sources (E = 662 keV) distributed on the ground to a depth of 5 g cm^{-2}, a value of 1.8 ± 0.2 may be used. However, if the sample is taken from the corner of the building, or the corner of a projection, the value of C_{RL} is expected to differ and simulations for the specific building geometry are necessary.

When determining D_T and calculating C_{RL}, an assessment of the potential contribution to the absorbed dose D_T by low energy photons (< 100 keV) is also required because experimental determinations of dose by luminescence are performed using $^{90}Sr/^{90}Y$ beta sources that are calibrated against a secondary standard ^{137}Cs or ^{60}Co photon source. As mentioned above and argued previously,[9] published data concerning fallout inventories from the tests,[20] indicate that the time-averaged mean source energy for fallout from the 1949 Semipalatinsk tests is within the range 500–800 keV. Although an experimental depth-dose profile cannot be used to unambiguously reconstruct source energy because it is a function of both energy and geometry,[14,24] those presented in this paper support such an interpretation if it is assumed that the fallout was primarily distributed on the ground. The proportion of absorbed dose arising from photons of energy < 100 keV is consequently likely to be small. Also, no further correction to the calculation of D_{BG} is required since less than 5% of the total energy emitted by the naturally occurring radionuclides is carried by photons of energy less than 100 keV.[25]

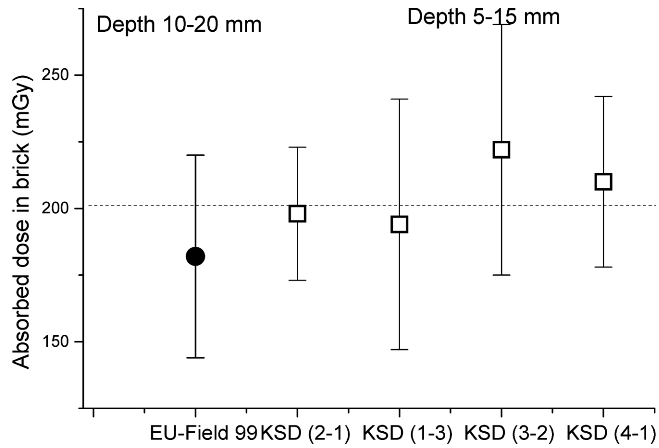

Fig. 4. Comparison of fallout dose in brick collected at a height of 1 m above ground at Dolon' in 1999 by the EU supported Measurements Group to those obtained from bricks at unknown height distributed for the intercomparison measurements.

Finally it should be noted that as far as the radioactive trace of 29 August, 1949 nuclear test seems to be narrow near Dolon' village,[9,26,27] further application of retrospective luminescence dosimetry method will be useful for more correct dose estimations in this village.

CONCLUSION

The measured values of the cumulative total does D_T quoted by the four laboratories was found to be in agreement within ± 10%, and within these limits, no systematic difference between OSL and TL determinations was observed. After subtraction of the cumulative background dose, D_{BG}, from DT, for a depth of 10 mm from the brick surface, the mean fallout dose, D_X, for the four intercomparison locations sampled in Dolon' is 204 ± 38 (s.d. 13) mGy. This value is in a good agreement with previously published values of cumulative dose in brick at a depth of 10 mm from the brick surface (182± 38 mGy) reported[9] by the EU-supported study group for samples that were collected during 1999 from the Large Church in Dolon'.

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