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Optical ages obtained for samples collected near Immarna and Barton, western South Australia (Ooldea project)

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Summary

Optical dating was applied to 6 samples from dunes from the Ooldea region of western South Australia. Quartz grains were extracted from the samples, 2.4 eV (green) excitation was used and the 3.4 eV (UV) luminescence emission band measured. The optical ages obtained were:

BN1/70: 105 ± 8 ka BN3/1.2: 71 ± 8 ka IA1/2.5: 188 ± 14 ka IA2/60: 22 ± 3 ka
BN1/2: 197 ± 14 ka IA1/3.5: 215 ± 15 ka

The samples are clearly much older than had been anticipated.

Sample collection

Samples were collected from sites near Barton and Immarna in western South Australia; the provenances provided by M.Sheard are given in table 1.

	grid reference	map	depth
Barton			
BN1/70	290334 6616407	53J	70 cm
BN1/2	"	"	2.0 m
BN2/70	290375 6616447	"	70 cm
BN3/1.2	290329 6616394	"	1.2 m
Immarna			
IA1/1	214144 6625063	"	1.0 m
IA1/2.5	"	"	2.5 m
IA1/3.5	"	"	3.5 m
IA2/60	214115 6625071	"	60 cm
IA3/4	787291 6624899	52J	4.0 m
IA4/1	214437 6624877	53J	1.0 m
IA5/1	214404 6624837	"	1.0 m

In-situ gamma spectrometry was performed for each sample collected except IA3/4. Six samples were dated.

Sample preparation

Approximately 150 g of raw sand from each sample was repeatedly treated in a 10% HCl solution to dissolve the carbonates. The treated sample was then dried in a 60°C oven for 2-3 days and sieved to select the 180-250 μm grain size fraction for dating. For each sample, approximately 3 g of the 180-250 μm grains were treated in 10% HF solution for 40 minutes to dissolve the feldspar fraction. A density separation was then performed using 2.9 g/ml sodium polytungstate solution to remove zircons and other dense minerals. A magnetic separation was performed and the non-magnetic fraction was selected for dating. We note that very little mass was lost during the etch and subsequent density and magnetic separations (<10% loss). Approximately 2.7-2.8 g of quartz grains were obtained for each sample.

Aliquots were prepared by evenly spreading 20-30 mg of the grains on 1.3 cm diameter aluminum planchets in which a few drops of 500 cSt silicone oil had previously been spread.

Measurement apparatus

Luminescence measurements were performed using our automated sample measurement chamber "Robot". Luminescence was detected using a Thorn-EMI[1] 9635QA photomultiplier tube and the UV quartz emission was selected using two 7-59, a 7-37 and BG-39 filters placed in series between the PMT and the sample[2]. This combination of filters had a 35 nm wide transmission band centred at 3.4 eV (370 nm). The excitation source consisted of a bank of 48 Nichia NSPG-500 high efficiency green light-emitting diodes fitted with individual GG-475 filters to absorb any emission above 2.76 eV (450 nm)[3]. These diodes provided approximately 8 mW/cm^2 of radiant power at the sample when operated at a current of 30 mA. These diodes have a 50 nm wide emission peak centered at 2.4 eV (525 nm).

Procedural details

Both additive dose (with thermal transfer correction) and regeneration measurements were done. Bleaching was performed under a car headlamp operated at fixed current near 12 V. A bleaching experiment using two planchets of each sample indicated that a 30 minute exposure to this light source resulted in roughly a hundred-fold decrease in the optically stimulated luminescence intensity in all samples. The bleach used in our measurements consisted of two 30 minute bleaches under the car headlamp separated in time by ~24 hours. The purpose of the repeated bleaching was to minimize the "recuperation" effect.

Sample irradiations were performed using a ^{60}Co gamma source and samples were preheated at 160°C for 16 hours (Huntley, Short and Dunphy, 1996) before measurement. We waited at least 24 hours between bleaching and irradiation, and irradiation and preheat.

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[1] Now "Electron Tubes Ltd".

[2] The filters were placed in the following order; nearest to the PMT: BG-39, followed by a 7-59 a 7-37 and a 7-59. This order is important because either the 7-37 or 7-59 fluoresce when exposed to the scattered light from the green diodes.

[3] Galloway and Neal (1998) discuss the use of these diodes in dating quartz.

Dosimetry

Table 2 gives the K, U and Th contents obtained from *in-situ* γ spectroscopy, K contents obtained from X-ray spectroscopy and U contents obtained by DNA analysis. We note that there is good agreement between the potassium contents from XRS and *in-situ* γ spectroscopy. Uranium contents from delayed neutron analysis (DNA) and from *in-situ* γ spectroscopy are not in such good agreement; at 1σ, values for sample BN3/1.2 almost overlap, however values for sample BN1/2 only agree within 2σ. This is not considered cause for concern since both techniques are being used near the lower limit of resolution.

sample	water	K (XRS)	U (DNA)	---------- *in-situ* γ-spectrometry[2] ----------		
	Δ[1]	%	$\mu g \cdot g^{-1}$	%	$\mu g \cdot g^{-1}$	$\mu g \cdot g^{-1}$
Barton						
BN1/70	0.023	0.096	0.30 ± 0.07	0.090 ± 0.007	0.34 ± 0.06	1.73 ± 0.11
BN1/2	0.017	0.115	0.56 ± 0.08	0.115 ± 0.007	0.34 ± 0.06	1.60 ± 0.11
BN2/70	0.022	0.195	0.38 ± 0.07	0.202 ± 0.009	0.63 ± 0.07	2.60 ± 0.13
BN3/1.2	0.025	0.068	0.24 ± 0.07	0.074 ± 0.007	0.39 ± 0.06	1.49 ± 0.10
Immarna						
IA1/1	0.017	0.107	0.23 ± 0.07	0.118 ± 0.007	0.31 ± 0.06	1.42 ± 0.10
IA1/2.5	0.027	0.100	0.30 ± 0.07	0.108 ± 0.007	0.33 ± 0.06	1.54 ± 0.11
IA1/3.5	0.021	0.123	0.48 ± 0.08	0.128 ± 0.007	0.39 ± 0.06	1.74 ± 0.11
IA2/60	0.023	0.083	0.22 ± 0.07	0.087 ± 0.007	0.33 ± 0.06	1.36 ± 0.10
IA4/1	0.063	0.349	1.84 ± 0.12	0.345 ± 0.014	2.31 ± 0.11	3.31 ± 0.15
IA5/1	0.078	0.275	0.81 ± 0.09	0.266 ± 0.011	1.02 ± 0.09	3.63 ± 0.15

Table 2: Water, potassium, uranium and thorium contents. [1]Water content is expressed as Δ = water mass divided by dry mass. [2]*In-situ* γ-spectrometry values have been corrected to dry values by multiplying the measured values by $(1 + 1.11*\Delta)$.

Thick-source alpha counting was performed on most of the samples and the results are given in Table 3. Comparison of these U and Th values with those in Table 2 shows that there is generally satisfactory agreement, an indication that radioactive disequilibrium is not a serious problem. The results of an alternative method of deducing Th contents are also shown in the table and there is a suggestion in them of a systematic difference.

sample	U (µg•g-1)	Th (µg•g-1)	Th (µg•g-1)
BN1/70	0.41 ± 0.09	1.48 ± 0.29	1.85
BN1/2	0.58 ± 0.05	1.25 ± 0.17	1.31
BN2/70	0.41 ± 0.08	2.99 ± 0.27	3.09
BN3/1.2	0.34 ± 0.09	1.66 ± 0.32	1.99
IA1/1	0.31 ± 0.09	1.74 ± 0.29	2.02
IA1/2.5	0.36 ± 0.09	1.57 ± 0.31	1.79
IA1/3.5	0.46 ± 0.13	1.58 ± 0.43	1.50
IA2/60	0.29 ± 0.09	1.34 ± 0.30	1.57
IA4/1	2.41 ± 0.30	2.53 ± 0.96	4.50
IA5/1	1.00 ± 0.08	3.49 ± 0.25	4.15

Table 3: U and Th contents determined by thick-source alpha counting. The last column is a thorium content calculated from the thorium contribution to the alpha count rate; the latter was obtained by calculating the uranium contribution to the alpha count rate using the uranium content determined by DNA (table 2), and subtracting it from the total alpha count rate.

Radiation dose rates were calculated using the dose conversion factors given by Nambi and Aitken (1986) as updated by Adamiec and Aitken (1999). The beta attenuation factor was calculated using the figures of Mejdahl (1979), assuming an average grain size of 215 µm. The beta dose rate from K, U and Th inside the grains was assumed to be negligible, however a total internal and external alpha dose rate of 0.03 ± 0.01 Gy/ka was assumed based on estimates made for quartz from the south-east of South Australia by Huntley *et al.* (1993a). Cosmic ray dose rates were calculated using the prescription of Prescott and Hutton (1994) and assuming a density of 2.0 g•cm^{-3}. No corrections were made for the change of sediment depth over time in computing the cosmic ray and gamma ray contributions. Dose rates were calculated in two ways, as independently as possible. The results are shown in Table 4.

		------ total dose rate -----	
sample	cosmic-ray	method A	method B
	Gy•ka^{-1}	Gy•ka^{-1}	Gy•ka^{-1}
		± 0.02	± 0.02
Barton			
BN1/70	0.191	0.497	0.501
BN1/2	0.160	0.482	0.465
BN3/1.2	0.178	0.465	0.458
Immarna			
IA1/2.5	0.150	0.457	0.459
IA1/3.5	0.132	0.486	0.486
IA2/60	0.193	0.470	0.455

Table 4. The calculated cosmic-ray dose rate, and the total dose rates calculated using two different combinations of the analytical results given in table 2. For method A, the in-situ γ-spectrometry values for K, U and Th were used. For method B, the K, U and Th values used were from XRS, DNA and thick-source alpha counting analyses respectively.

Discussion of the Equivalent doses and ages

Measurements for two of the samples, BN3/1.2 and IA2/60 were straightforward and yielded optical ages of 74 ± 8 ka and 22 ± 3 ka respectively; these are listed in Table 5. We did not perform a regeneration experiment for these samples.

Sample	Equivalent dose, Gy	Dose rate Gy/ka	Age, ka
BN3/1.2	33 ± 3	0.462 ± 0.02	71 ± 8
IA2/60	10 ± 1.5	0.463 ± 0.02	22 ± 3

Table 5: Equivalent doses, dose rates and ages obtained for the youngest samples.

The four other samples BN1/70, BN1/2, IA1/2.5 and IA1/3.5 presented problems, the luminescence intensities of the natural, untreated aliquots for these samples were 40% or more up the saturation growth curve. This was unexpected since we had expected the samples to be no older than the last glacial period. Regeneration experiments in which data was taken well into the saturation region were therefore performed. The Australian slide procedure was used to analyze the data. In this, the additive-dose data is shifted along the dose axis and the best match to the regeneration data found. The shift is taken as the equivalent dose after correcting for the decay during the normalization measurement. To find the best match the data were fitted to a function that describes the dose response. Here we assumed that the luminescence intensity **I** as a function of applied dose **D** followed a saturating exponential of the form,

$$\mathbf{I = I_{sat}\,(1 - e^{-D/Dc})},$$

where **I** is the luminescence intensity, **D** is the radiation dose, $\mathbf{D_c}$ is a characteristic dose that determines the onset of saturation and $\mathbf{I_{sat}}$ is the saturation intensity. The data and fit for IA1/3.5 are shown in figure 1. The equivalent doses and ages determined from them are shown in Table 6.

Sample	Equivalent dose, Gy	Dose rate Gy/ka	age ka
BN1/70	52.6 ± 3.5	0.499 ± 0.02	105 ± 8
BN1/2	93.3 ± 5.9	0.474 ± 0.02	197 ± 14
IA1/2.5	86.3 ± 5.1	0.458 ± 0.02	188 ± 14
IA1/3.5	104.6 ± 6.2	0.486 ± 0.02	215 ± 15

Table 6: Equivalent doses and ages for the older samples.

The dose-response curves show that these samples are approaching saturation (see for example figure 1), and three of the equivalent doses are over 85 Gy. To our knowledge, no one has determined whether or not equivalent doses of over 60 Gy yield correct ages for quartz. We therefore tested the assumption that "old" quartz is indeed in saturation using four samples from elsewhere in southern Australia. The results are described in the Appendix and show that for these samples they are typically at 96% of the saturation intensity; because of the scatter we cannot be sure that they are not at 100%. This test would be better had it been done on samples from the Ooldea region, but such samples were not available. We can only say that it is likely that quartz from different regions behaves similarly and on this assumption we conclude that the ages may be slightly older than those shown in Table 4. If the 96% figure is valid the ages should be increased by 4% for BN1/70 and 7% for the others. The significance of this is that although

the ages of BN1/2, IA1/2.5 and IA1/3.5 correspond to $\delta^{18}O$ stage 7, one should not attempt to allocate them to any particular substages.

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Appendix

The purpose of this work was to answer the question of whether or not, under natural conditions in which the past environmental radiation dose is large, say 500 Gy or more, and in the context of the present work, quartz is saturated. As will be seen the answer appears to be that it is within about 5% of saturation. The reason for being concerned about this point is that under comparable circumstances feldspars are not saturated.

Four samples were studied. All are from the south-eastern part of South Australia and neighboring Victoria, and were chosen because separated quartz grains were already available. The samples were:

KV1, from a dune near Kaniva, Victoria, thought to be Tertiary in age.

SESA-105, from the Marmon Jabuk Range, thought to be Tertiary in age.

SESA-146, from the East Naracoorte dune near Naracoorte, specifically from the municipal garbage dump. Current matching of the dune system to high sea stands places the age at about 0.9 Ma.

SESA-46, from the Baker Range. The age of this dune can be confidently stated to be very close to 0.48 Ma as a result of our thermoluminescence dating work and the matching of the dune sequence to high sea stands. This sample was not expected to be in saturation, but close to it.

The four samples were subjected to regeneration measurements in the same way that the Ooldea ones were. The doses were chosen to establish the saturation intensites for both data sets, and as well to provide values of D_c, the characteristic dose for the saturating exponential.

The data were fitted with our regeneration software. This procedure is not expected to produce a useful dose shift for a sample that is in saturation. Figure 2 shows the data and fit for KV1; no value should be placed on the dose shift shown. The data for SESA-105 are rather more scattered than those of the others and not much reliance shuld be placed on them.

In order to provide numerical figures for the fraction of the saturation intensities, the following intensity ratios were worked out

Sample	I (N) / I(N+dose)	I(N) / I(N+bleach+large dose)
KV1	0.966 ± 0.034	0.979 ± 0.033
SESA-105	0.945 ± 0.067	0.883 ± 0.038
SESA-146	0.944 ± 0.028	0.976 ± 0.039
SESA-46	0.818 ± 0.061	0.803 ± 0.040

By large dose is meant 450 Gy or more.

The normalization measurement empties a small fraction of the traps. Analaysis of the data shows that the luminescence decreased by 1.0% during this measurement. For proper analysis this needs to be corrected for.

Values for D_c, the characteristic dose of the saturating exponential, obtained using joint fits to both data sets in the usual fashion, were:

KV1	105 ± 4 Gy
SESA-105	113 ± 12 Gy
SESA-146	102 ± 5 Gy
SESA-46	104 ± 6 Gy

The best estimate would appear to be 104 ± 3 Gy. In the following this is used to estimate the fraction of the saturation value that is expected for the above samples on the assumption that the dose response is precisely a saturating exponential with the above value of D_c.

The following table shows a comparison of I(N) / I(N+dose), after correction for the 1% decrease during the normalization measurement, with the the value expected on the basis of the above D_c, age and dose rate.

sample	age (Ma)	dose rate (Gy/ka)	D_{eq} (Gy)	ratio expected	ratio measured
KV-1	≥ 2	1.1	≥ 2200	1.000	0.976 ± 0.034
SESA-105	≥ 2	1.2	≥ 2400	1.000	0.955 ± 0.067
SESA-146	~ 0.9	0.62*	560	0.995	0.954 ± 0.028
SESA-46	0.48	0.48	230	0.890	0.826 ± 0.061

* from G.B.Robertson

It is seen that the measured ratios are typically 4% less than expected on the basis of the calculation. The scatter in the data is more than it should be and it would be unwise to conclude that there really is a 4 % effect.

It will be noticed that the points labelled "Nnp" on figure 1 are not on top of each other as they should be; this was also true for the other samples. This indicates that there is something about the experimental technique that introduced an uncontrolled source of scatter; we do not normally observe this and it could presumably be much reduced if future measurement were to be made. The working hypothesis is that the normalization measurements were made on the same day that the planchets were prepared, and that the quartz grains moved afterwards.

IA1/3.5

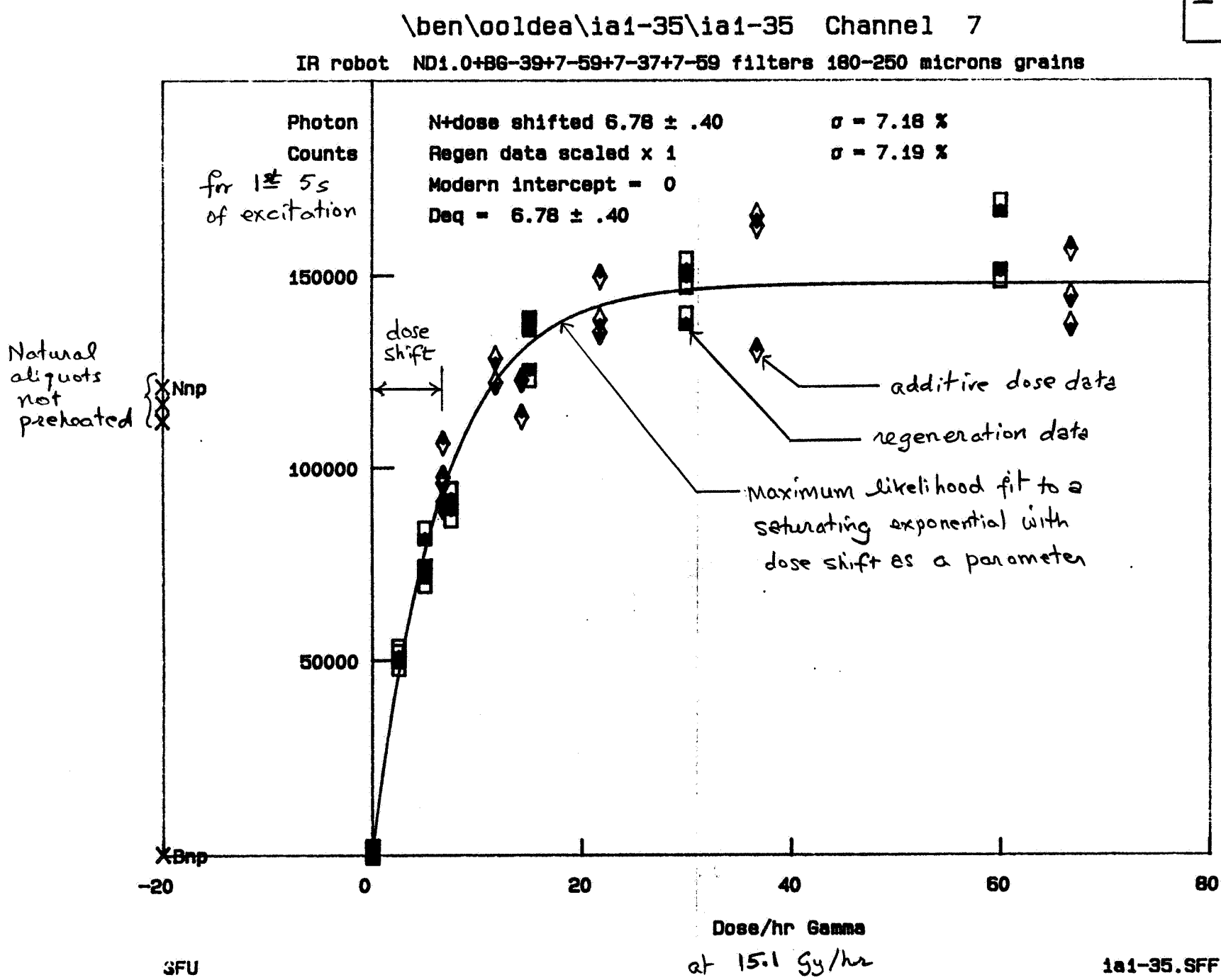
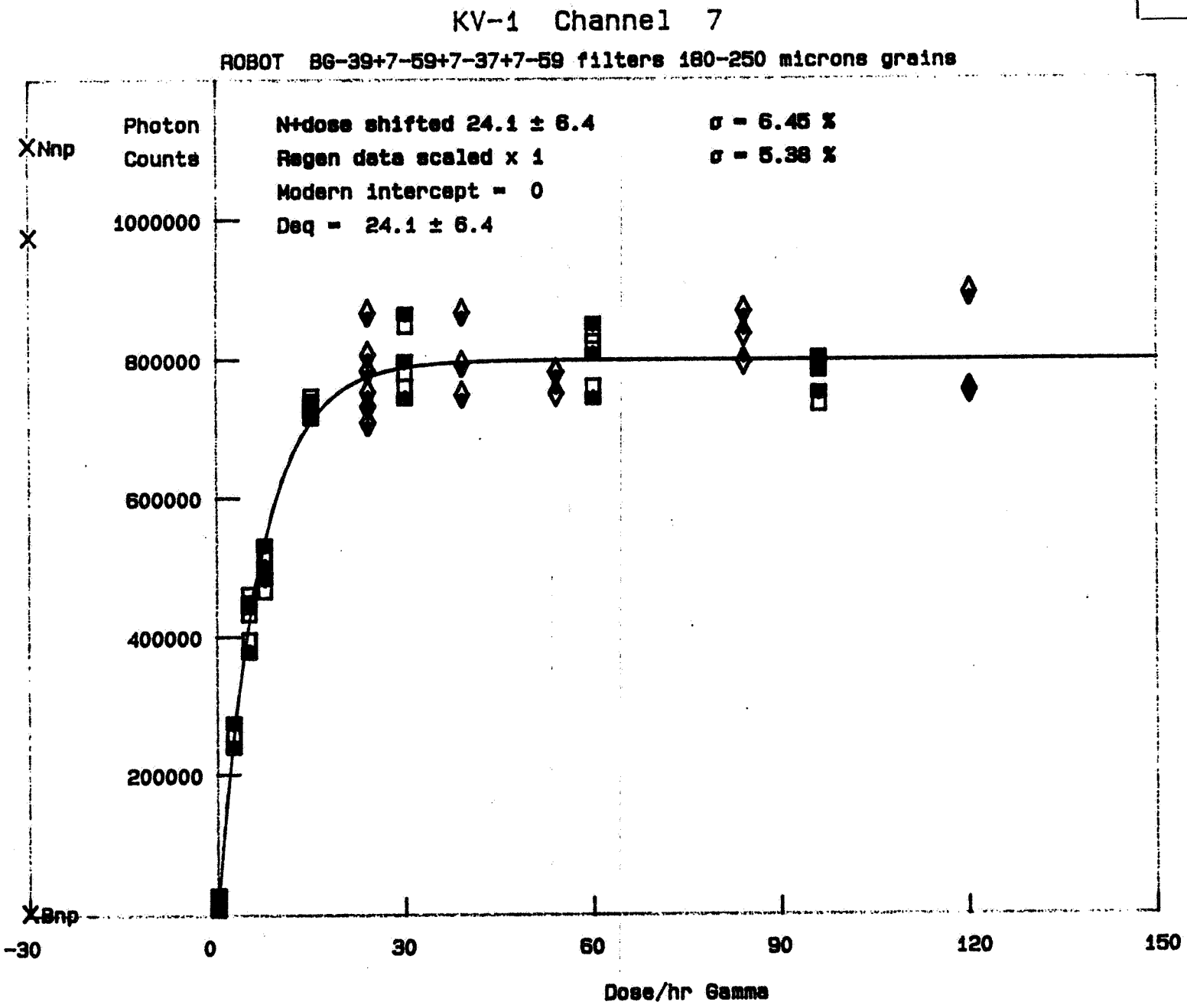

\ben\ooldea\ia1-35\ia1-35 Channel 7
IR robot ND1.0+86-39+7-59+7-37+7-59 filters 180-250 microns grains
Photon Counts
for 1st 5s of excitation
N+dose shifted 6.78 ± .40
σ = 7.18 %
Regen data scaled x 1
σ = 7.19 %
Modern intercept = 0
Deq = 6.78 ± .40
150000
100000
50000
Natural aliquots not preheated
Nnp
Bnp
dose shift
additive dose data
regeneration data
Maximum likelihood fit to a saturating exponential with dose shift as a parameter
-20
0
20
40
60
80
Dose/hr Gamma
at 15.1 Gy/hr
SFU
ia1-35.SFF
plotted 03-23-1999

FIG. 1

KV-1



KV-1.SFF
plotted 04-27-1999

Fig 2