CHAPTER III

EXPERIMENTAL RESULTS AND DISCUSSION

3.1 Radiometric method

3.1.1 Direct determination of ThO_2 by γ-activity measurement under the 2.61 Mev and the 0.91 Mev peaks The determination of ThO_2 by γ-activity measurement under the 2.61 Mev peak was first checked by 3 different detectors, namely A, B and C. Since it was found that the results from the 3 detectors agree well with one another, the activity under the 0.91 Mev peak was measured via detector A only. The conditions of the counters were set as follows:

Detector A: amplifier gain 2, window width 2,

Detector B: amplifier gain 6, zero level 8,

Detector C: coarse gain 20, fine gain 1500, conversion gain 1024.

The calibration curves for the three detectors at various analyser resolutions are shown in Figures 3.1, 3.2 and 3.3.

FIGURE 3.1 CALIBRATION CURVE FOR THE DETECTOR A

a) GAIN 2, WINDOW WIDTH 2,

b) GAIN 8, WINDOW WIDTH 2

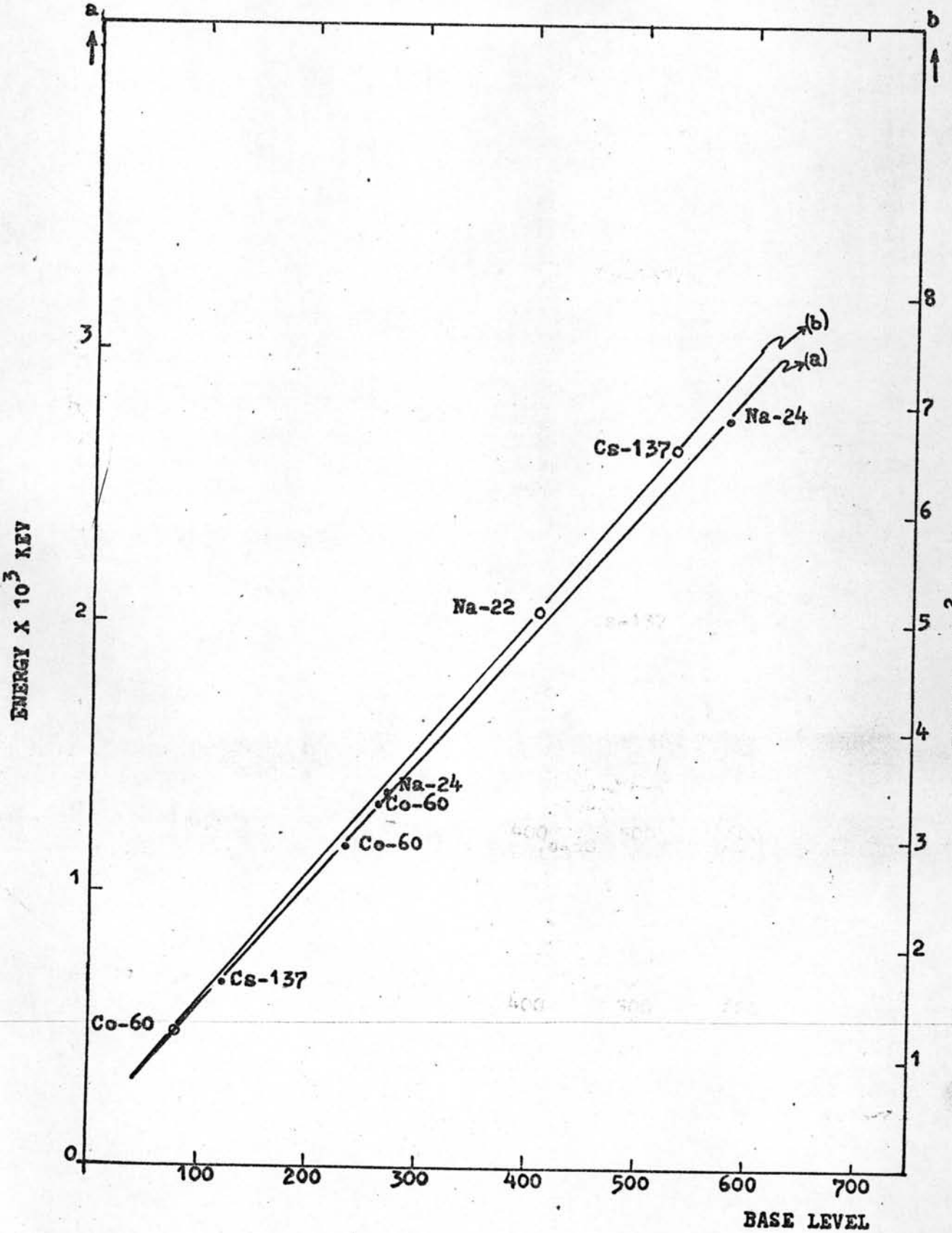


FIGURE 3.2 CALIBRATION CURVE FOR THE DETECTOR B AT GAIN 6, ZERO LEVEL 8

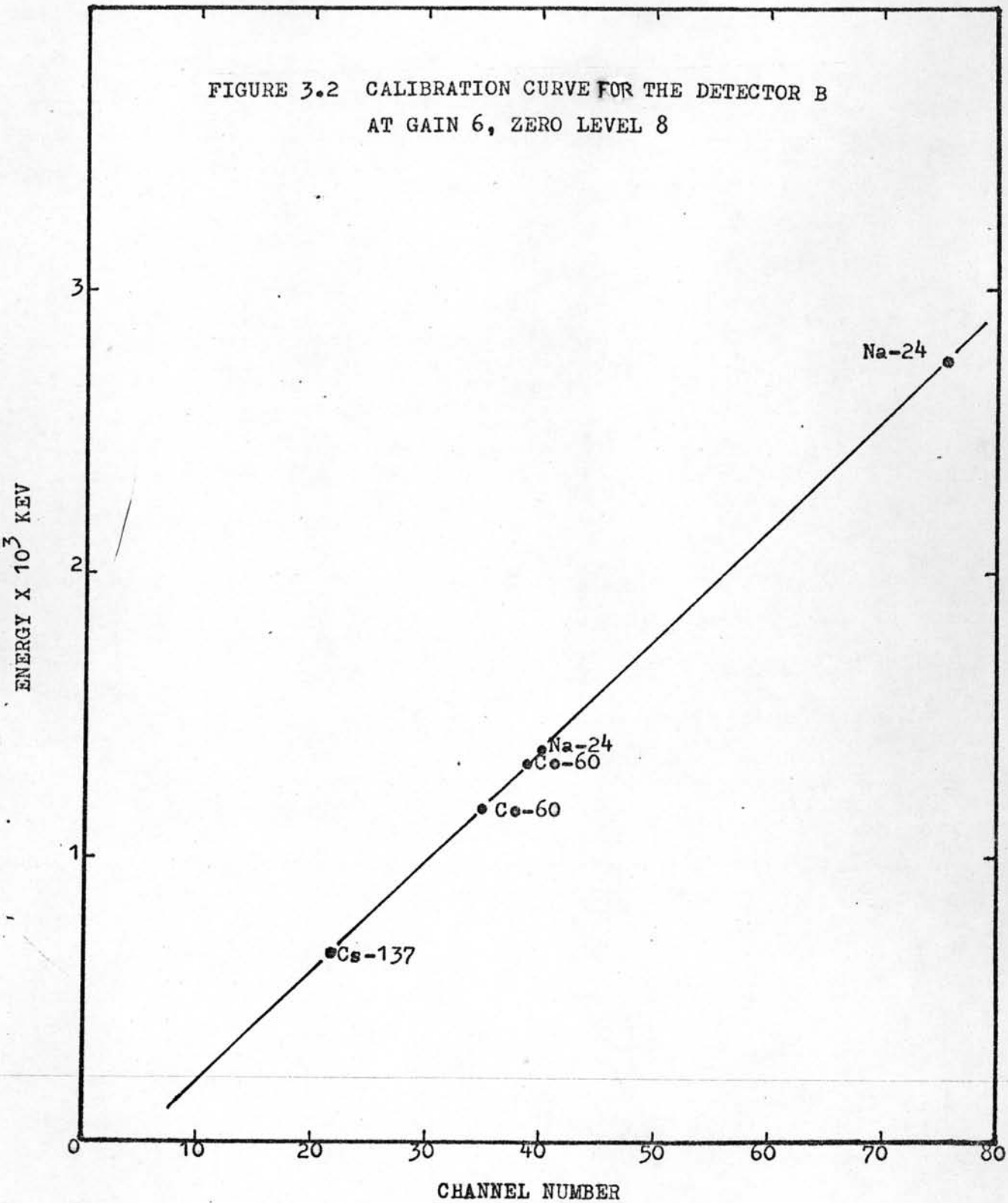
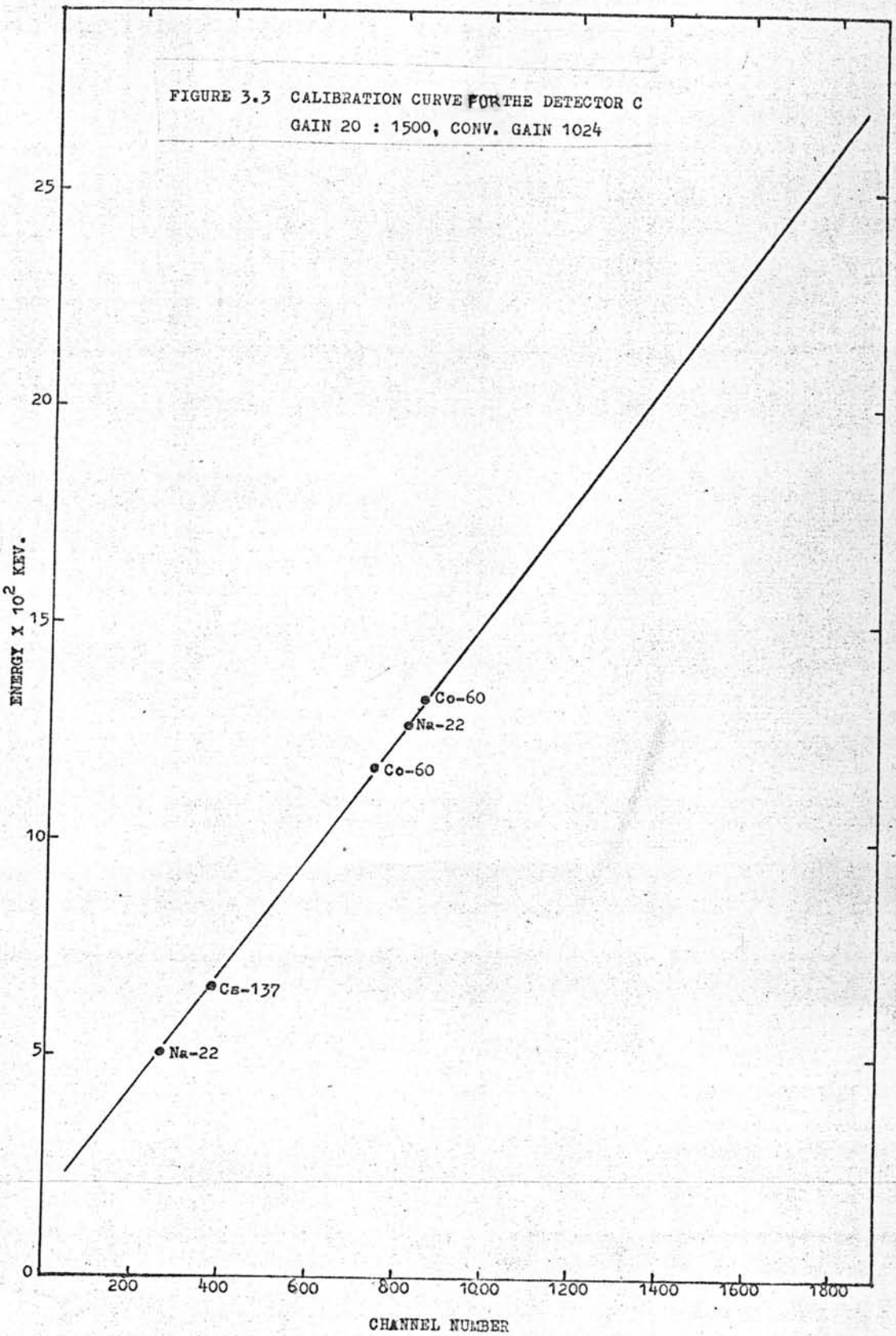


FIGURE 3.3 CALIBRATION CURVE FOR THE DETECTOR C
GAIN 20 : 1500, CONV. GAIN 1024



Since the counting efficiency of the detectors are not similar, the time of activity measurement varied so that good counting statistics could be obtained. Normally the counting time was 10 minutes for detector B, 4000 sec for detector C; 30 minutes for detector A when the sample container was a planchette and 20 minutes when the sample container was a polyethylene vial. The spectra of the monazite NBL-7A, of one local monazite and of the uranium ore S_1 as obtained from detector A, B and C are shown in Figures 3.4, 3.5 and 3.6 respectively.

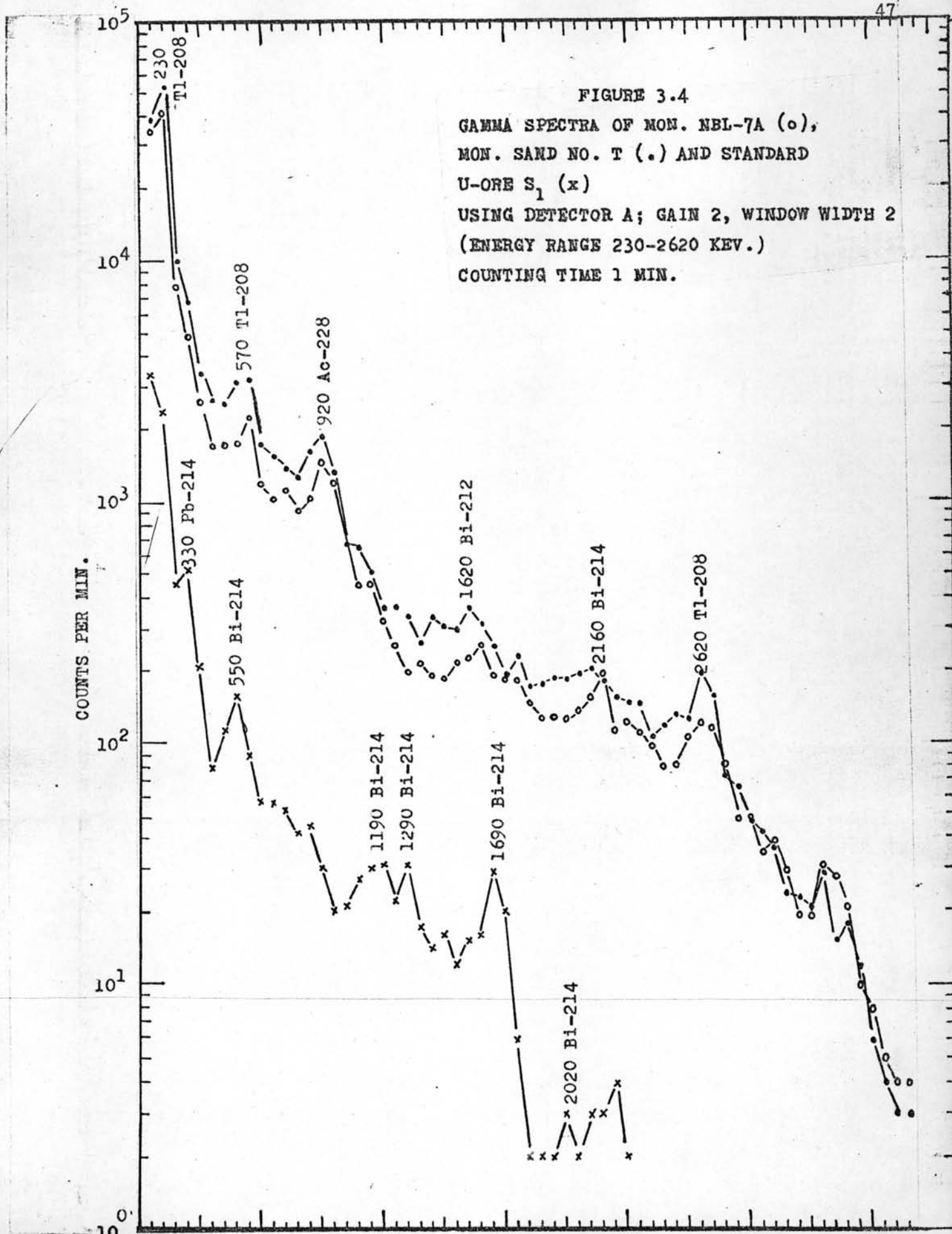


FIGURE 3.4

GAMMA SPECTRA OF MON. NBL-7A (o), MON. SAND NO. T (.) AND STANDARD U-ORE S_1 (x)
USING DETECTOR A; GAIN 2, WINDOW WIDTH 2
(ENERGY RANGE 230-2620 KEV.)
COUNTING TIME 1 MIN.

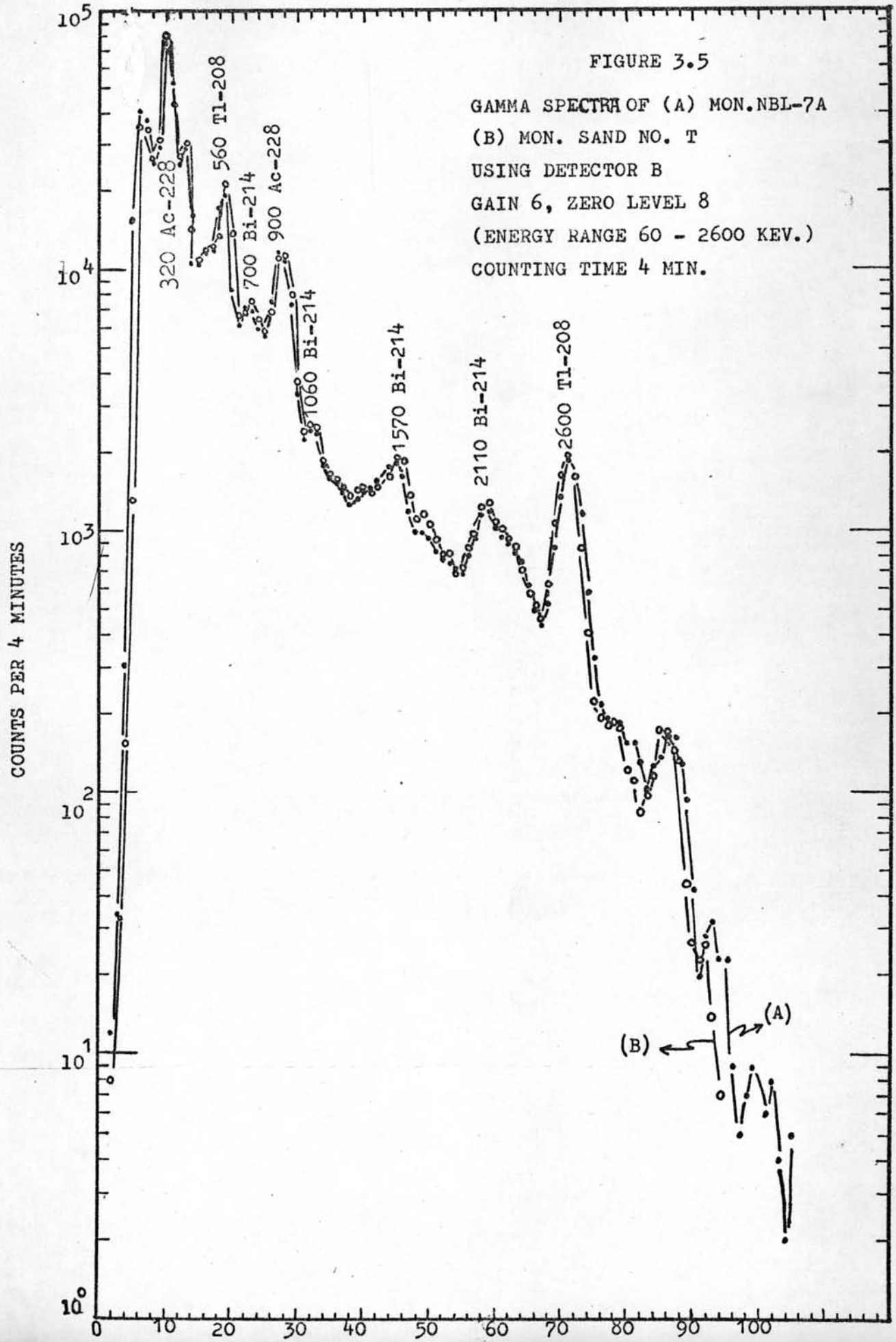


FIGURE 3.5

GAMMA SPECTRA OF (A) MON.NBL-7A
(B) MON. SAND NO. T
USING DETECTOR B
GAIN 6, ZERO LEVEL 8
(ENERGY RANGE 60 - 2600 KEV.)
COUNTING TIME 4 MIN.

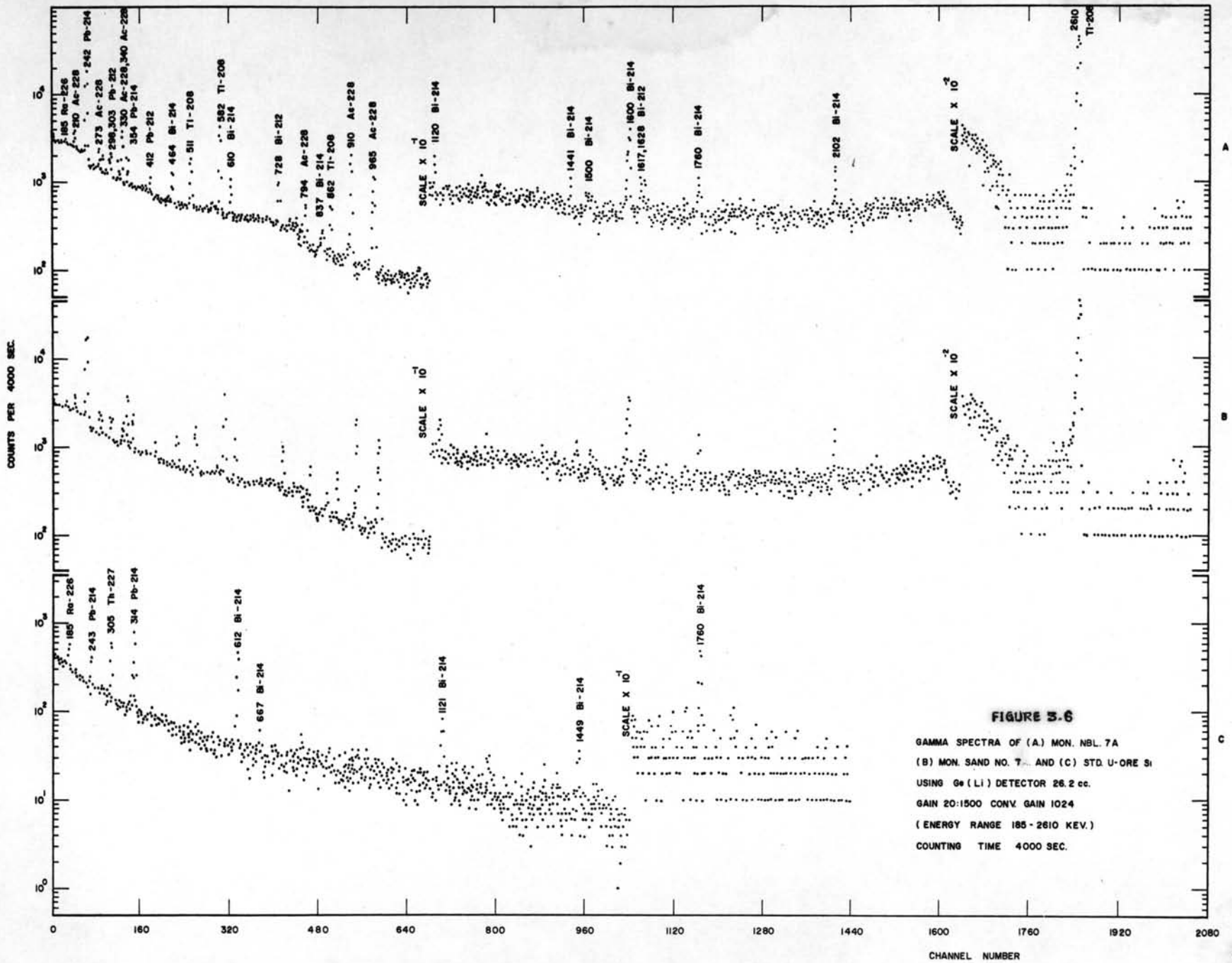


FIGURE 3.6

GAMMA SPECTRA OF (A.) MON. NBL. 7A
(B) MON. SAND NO. 7 AND (C) STD. U-ORE SI
USING Ge(Li) DETECTOR 26.2 cc.
GAIN 20:1500 CONV. GAIN 1024
(ENERGY RANGE 185 - 2610 KEV.)
COUNTING TIME 4000 SEC.

Table 3.1

ThO_2 contents in monazite sample from intensity measurement at 2.61 Mev.

sample	wt. (gm)	Detector A			Detector B			Detector C			Detector A (Sample in polyethylene vial)			
		cpm	% Th	$\%ThO_2$	cpm	% Th	$\%ThO_2$	Act/4000 sec.	% Th	$\%ThO_2$	wt. of sample (gm)	cpm	% Th	$\%ThO_2$
Mon. NBL-7A	11.72525	52.00	(8.52)	(9.7)	1493.0	(8.52)	(9.7)	2207.0	(8.52)	(9.7)	4.04970	103.30	(8.52)	(9.7)
1	12.86170	35.37	5.29	6.02	1101.5	5.73	6.52	-	-	-	4.33915	69.80	5.37	6.12
T	16.17780	79.27	9.49	10.72	2132.5	8.82	10.05	3151.0	8.74	9.93	5.11210	120.80	7.89	8.99
G_1	16.59370	-	-	-	1549.9	6.25	7.12	-	-	-	5.08235	87.20	5.73	6.52
G_2	15.11015	-	-	-	1542.3	6.83	7.78	-	-	-	4.74280	81.85	5.93	6.56
B	13.75140	46.07	6.44	7.33	1223.1	5.95	6.78	-	-	-	4.78760	75.35	5.26	6.50
6.1	12.26555	38.60	6.05	6.89	1041.2	5.68	6.48	1577.0	5.77	6.56	4.93125	84.85	5.75	6.54
7	12.96362	51.77	7.68	8.74	1433.7	7.40	8.44	2183.5	7.40	8.41	5.14420	105.50	6.85	7.80
9	14.31908	41.60	5.58	6.36	1204.9	5.65	6.42	-	-	-	5.12920	82.95	5.40	6.15
10	15.24835	50.13	6.32	7.19	1465.3	6.43	7.33	-	-	-	5.35605	92.25	5.75	6.55
11.1	13.86610	42.67	5.91	6.73	1340.7	6.47	7.38	-	-	-	5.02470	90.10	5.99	6.82
11.2	13.69923	45.97	6.45	7.07	1402.4	6.85	7.81	-	-	-	4.99500	88.55	5.92	6.74
A	13.17942	47.13	6.87	7.82	1404.4	7.13	8.13	1881	6.46	7.35	4.95230	92.20	6.22	7.08

Note: 1. - not measured

2. All activity measurements were made for 30 min (sample in planchette) and 20 min (sample in vial) using detector A, 10 min using detector B and 4000 sec. using detector C.

From Table 3.1, in which the results for the determination of ThO_2 contents by intensity measurements at 2.61 Mev are presented, it could be observed that the results from various detectors are in good agreement. Detector A which is connected to the most simple analyser, the single channel analyser, gives agreeable results in reasonably short time. From the results of the thorium contents in three standard thorium ores, Table 3.2, it was found that the thorium content of as low as 0.05% can be determined accurately by the simple single channel analyser.

Table 3.2

Thorium contents in standard thorium ores by γ-activity measurements at 2.61 Mev peak.

Standard	wt.(gm)	Activity/30 min.	Activity per 30 min. per gm.	% Th (experiment)	% Th (expected)
Mon. NBL-7A	4.0497	2345.0	579.1	-	(8.52)
S - 79 A	2.4924	177.0	71.0	1.040	1.00
S - 80 A	2.7377	20.5	7.5	0.110	0.1005
S - 81 A	2.7802	10.0	3.6	0.053	0.05

The results of the determination of the thorium contents by γ-activity measurement under the 0.91 Mev peak using detector A are shown in Table 3.3. Since the activity contributions of uranium were neglected in the evaluation, the results of the ThO_2 contents

should be higher than the actual values. However, they agree within 10% with those obtained by γ-activity measurement under the 2.61 Mev peak when the same detector (detector A) is used.

Table 3.3

Thorium content from γ-ray measurement at 0.91 Mev using detector A

Sample	wt. (gm.)	Act. after backg. subtraction		Concentration (not corrected for uranium contribution)				%Th by NAA
		1st run (c/5 min)	2nd run (cpm)	1st run		2nd run		
				% Th	$\%ThO_2$	% Th	$\%ThO_2$	
Mon.NBL-7A	4.04970	5873	1240.2	(8.52)	(9.7)	(8.52)	(9.2)	(8.52)
S_1	1.89890	167	33.6	-	-	-	-	-
1	4.33915	4129	862.8	5.85	6.66	5.78	6.58	6.18
T	5.11210	7662	1625.2	9.21	10.49	9.24	10.52	7.16
G_1	5.08235	5651	1212.2	6.83	7.78	6.93	7.89	6.26
G_2	4.74280	5530	1164.0	7.17	8.16	7.13	8.12	5.76
B	4.78760	4965	1037.2	-	-	5.07	5.78	5.76
6.1	4.93125	5159	1056.6	6.43	7.32	6.23	7.09	5.64
7	5.14420	6778	1435.8	8.10	9.22	8.11	9.24	7.28
9	5.12920	5326	1096.8	6.38	7.27	6.22	7.08	5.52
10	5.35605	6117	1276.6	7.02	7.99	6.93	7.89	6.52
11.1	5.02470	5772	10470	-	-	6.06	6.90	6.56
11.2	4.99500	6029	1276.8	7.42	8.45	7.43	8.46	7.40
A	4.95230	6088	1240.2	7.56	8.60	7.28	8.29	6.36

Note : 1. Values in brackets are reference values.

2. Activity of thorium in Mon.NBL-7A after correction of uranium contribution are 1121.68 and 1186.96 cpm for the first and the second runs, respectively.

3.1.2 Equilibrium conditions of samples

3.1.2.1 Method based on the measurements of gross β - and gross γ-activity. The gross β - and gross γ-activities were measured using the G.M. counter and detector A, respectively. The counting time per sample was normally 10 minutes for the G.M. counter and 1 minute for detector A. Two sets of γ-measurements were performed. In the first case, the base line was adjusted so as the response from all radiations below 80 Kev were cut off and the analyser operated at gain 8 so that upto 1240 Kev can be measured. In the second case, the base line was adjusted so as the response below 340 Kev were cut off and the analyser operated at gain 2 so that upto 4700 Kev can be detected.

Table 3.4

Equilibrium condition of samples from $e(U_3O_8)_\beta$ and $e(U_3O_8)_\gamma$ measurements.

Sample	Wt.(gm)	Activity (cpm)	Sp.act. (cpm)	$\%U_3O_8$	$e(U_3O_8)_\beta$	Gross-γ measurement; integrate from 340 Kev				Gross-γ measurement; integrate from 80 Kev				$\frac{e(U_3O_8)_\beta}{e(U_{38})_{\gamma_I}}$	$\frac{e(U_3O_8)_\beta}{e(U_{38})_{\gamma_{II}}}$
						Activity (cpm)	Sp.act. (cpm)	$\%U_3O_8$	$e(U_{38})_{\gamma_I}$	Activity (cpm)	Sp.act. (cpm)	$\%U_3O_8$	$e(U_{38})_{\gamma_{II}}$		
MonNBL-7A	11.72525	346.87	29.58	-	3.42	14672.20	1251.33	4.57	9.70	41253	3518.30	4.11	8.73	0.35	0.39
S_1	6.61625	57.07	8.63	(0.471)	(1)	852.20	128.80	(0.471)	(1)	2663	402.49	(0.471)	(1)	(1)	(1)
S_2	5.32849	38.43	7.21	(0.313) 0.396	0.84	460.20	86.37	0.315	0.67	1407	264.05	0.309	0.67	1.25	1.25
S_3	6.71155	58.27	8.68	(0.418) 0.468	0.99	560.20	83.47	0.305	0.65	1996	297.40	0.348	0.74	1.52	1.34
S_4	5.55980	44.87	8.07	(0.375) 0.444	0.94	515.20	92.67	0.338	0.72	1675	301.27	0.352	0.75	1.31	1.25
1	12.86170	256.27	19.93	-	2.31	10881.20	946.02	-	7.32	29870	2322.40	-	5.77	0.32	0.40
T	16.17780	410.37	25.37	-	2.95	20331.20	1256.73	-	9.75	57842	3575.39	-	8.87	0.30	0.33
B	13.75410	267.07	19.42	-	2.27	12403.20	901.78	-	6.98	34861	2534.59	-	6.31	0.33	0.36
6.1	12.26555	288.57	23.53	-	2.74	11600.20	945.75	-	7.32	31337	2554.88	-	6.34	0.37	0.43
7	12.96362	353.37	27.26	-	3.18	15270.20	1177.93	-	9.13	43540	3358.63	-	8.34	0.35	0.38
9	14.31908	291.57	20.36	-	2.38	12475.20	871.23	-	6.75	34526	2411.19	-	5.99	0.35	0.40
10	15.24835	312.57	20.50	-	2.40	14978.20	982.28	-	7.62	41237	2704.36	-	6.71	0.31	0.36
11.1	13.86610	311.77	22.48	-	2.63	13594.20	980.39	-	7.60	39834	2872.76	-	7.13	0.35	0.37
11.2	13.69923	316.27	23.09	-	2.70	13453.20	982.04	-	7.60	39570	2888.48	-	7.18	0.36	0.38
R	13.17942	253.27	19.22	-	2.25	13991.20	1061.59	-	8.22	43540	3303.64	-	8.22	0.27	0.27

Note : All activities were not subtracted for counts of thorium.

Values in brackets are reference values.

Table 3.5

Equilibrium condition of samples from $e(U_3O_8)_\beta$ and $e(U_3O_8)_\gamma$ measurements.

BG = 881 cpm | BG = 255.8 cpm | BG = 75.3 c/10 min.

Sample	wt. (gm.)	concentration of ThO_2 for correction * (%)	gross γ-activity measurement (integrate from 80 Kev)					gross γ-activity measurement (integrate from 340 Kev)					gross β-activity measurement					ratio of $e(U_3O_8)_\beta$ to $e(U_3O_8)_\gamma$	
			activity after backg. subtraction	activity of U after corr. for ThO_2 (cpm)	sp. act. of U cpm/gm sample	% U_3O_8	$e(U_3O_8)_\gamma$	act. after backg. subt. (cpm)	contr. of U after corr. for ThO_2 (cpm)	sp. act of U cpm/gm sample	% U_3O_8	$e(U_3O_8)_\gamma$	act. after backg. subt. (cpm)	contrib. of U after corr. for ThO_2 (cpm)	cpm/gm	% U_3O_8	$e(U_3O_8)_\beta$	$e(U_3O_8)_\gamma$ from 80 Kev	$e(U_3O_8)_\gamma$ from 340 Kev
Hon.NBL-7A	11.72525	(9.7)	41253	3569.178	299.11	(0.35)	-	14672.20	1122.419	95.75	(0.35)	-	346.87	75.16	6.41	(0.35)	-	(1.00)	(1.00)
S_1	6.61625	-	2663	2663.00	402.49	(0.471)	-	852.20	852.20	128.80	(0.471)	-	57.07	57.07	8.63	(0.471)	-	(1.00)	(1.00)
1	12.86170	6.12	29870	3751.19	291.66	0.341	0.72	10881.20	1503.63	116.91	0.43	0.91	256.27	68.69	5.34	0.29	0.62	0.86	0.68
2	16.17780	8.59	57842	9586.92	592.60	0.693	1.47	20331.20	3004.49	185.72	0.68	1.44	430.37	63.78	3.94	0.22	0.47	0.31	0.33
3	13.75410	6.50	34861	5191.10	377.42	0.442	0.94	12403.20	1752.32	127.40	0.47	0.99	269.07	54.95	4.00	0.22	0.47	0.50	0.47
6.1	12.26555	6.54	31337	4220.38	384.85	0.450	0.96	11600.20	2043.57	166.61	0.61	1.29	288.57	97.40	7.94	0.44	0.93	0.92	0.72
7	12.56362	7.80	43540	9987.12	770.40	0.901	1.91	15220.20	3223.78	248.68	0.91	1.93	353.37	12.40	8.67	0.48	1.02	0.53	0.53
9	14.31908	6.15	34526	5287.54	369.27	0.432	0.92	12425.10	1983.96	138.55	0.51	1.08	291.57	81.72	5.70	0.31	0.66	0.72	0.61
10	15.24835	6.55	41237	8052.38	530.05	0.620	1.32	14978.20	3079.38	201.95	0.74	1.57	312.57	74.57	4.89	0.29	0.57	0.43	0.36
11.1	13.86610	6.82	39834	8438.33	608.56	0.712	1.51	13594.20	2327.99	167.89	0.61	1.29	311.77	86.42	6.23	0.34	0.72	0.48	0.56
11.2	13.69923	6.74	39570	8037.65	652.42	0.829	1.76	13453.20	2453.14	179.07	0.65	1.38	316.27	96.24	7.03	0.39	0.83	0.47	0.60
A	13.17942	7.08	43540	12075.77	954.20	1.116	2.37	13901.20	2824.74	218.32	0.80	1.70	253.27	31.23	2.37	0.13	0.28	0.12	0.16

Note : 1. Values in brackets are reference values.

2. (a) calculated from the activity of U-ore S_1.

3. *concentration of ThO_2 in percent from 2.61 Mev Measurement using detector A

4. Contribution of thorium in Hon. NBL-7A is 37745.83 cpm.

From Table 3.4 in which the results are presented it could be observed that the $e(U_3O_8)_\gamma$ values of the two series of measurements agree well with each other in the case of uranium ores (sample S_2, S_3 and S_4) and have a difference of about 15% in the case of monazite samples. Considering the errors in these measurements, the variations of the $e(U_3O_8)_\beta$ / $e(U_3O_8)_\gamma$ ratios of the uranium ores from 1 are not considered significant. Hence it is considered that the three uranium ores are in or nearly in radioactive equilibrium. In the case of monazite samples which contained both uranium and thorium, it was found that the ratios of $e(U_3O_8)_\beta$ / $e(U_3O_8)_\gamma$ lie between 0.3 and 0.4. Attempts were made to evaluate the contents of uranium in the monazite samples by gross activity measurements. The standard monazite NBL was corrected for the 0.4% U_3O_8 content using the standard U-ore S_1 data and the monazite samples were corrected for the thorium oxide content (from NAA) using the corrected monazite NBL data. The values thus obtained were then used for calculation which results are shown in Table 3.5. It could be observed that the results from gross β -measurements do not agree with the results from gross γ-measurements. The limitation of this method for the determination of the uranium contents in monazite samples might be caused by two reasons. First, the uranium family in the samples might not be in RE. Second, the high ThO_2: U_3O_8 ratio of the samples might cause large errors in the evaluation.

3.1.2.2 Method based on the γ-activity ratio at the 80 - 100 Kev and 610 Kev peaks. Detector A which amplifier was set at gain 8 and which was calibrated to give an energy of 1.2 Kev/base level

was used for the measurements. The spectra of the standard monazite NBL-7A, one monazite sample and the standard U-ore S_1 as measured by the mentioned set-up are shown in Figure 3.7.

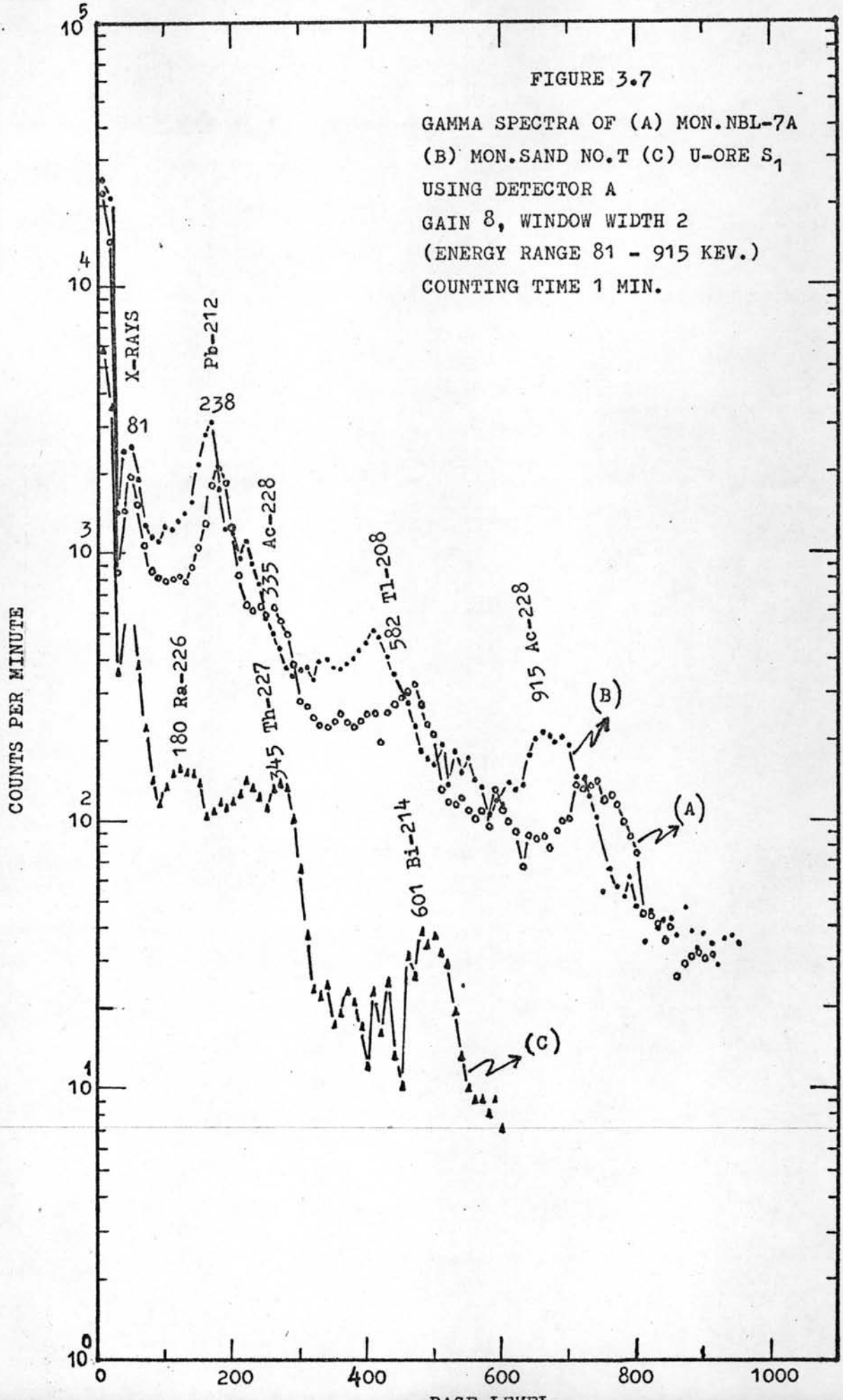


FIGURE 3.7
GAMMA SPECTRA OF (A) MON.NBL-7A
(B) MON.SAND NO.T (C) U-ORE S_1
USING DETECTOR A
GAIN 8, WINDOW WIDTH 2
(ENERGY RANGE 81 - 915 KEV.)
COUNTING TIME 1 MIN.
COUNTS PER MINUTE
BASE LEVEL
X-RAYS
81
Pb-212
238
180 Ra-226
345 Th-227
335 Ac-228
582 Tl-208
915 Ac-228
601 Bi-214
(A)
(B)
(C)

As was performed in section 3.1.2.1, the thorium contents in the monazite samples were corrected using the monazite NBL-7A which 0.35% U_3O_8 content was already corrected using the U-ore S_1. Since the detector efficiency at 610 Kev is lower than the efficiency at 80 - 100 Kev by a factor of 3 (see Figure 2.1 a), all count rates obtained from measurement at 610 Kev were multiplied by 3. The results of the experiment are shown in Table 3.6 a) and b). Considering the error in these measurements, the variations of the activity ratios between those for uranium ores (samples S_2, S_3, S_4) and that for the standard U-ore S_1 are not considered significant. Hence the results of the present method agree well with the results from the measurements of the gross β /gross γ ratio that the uranium-ore samples S_2, S_3 and S_4 are in or nearly in radioactive equilibrium. In the case of monazite samples, it was found that all ratios except that from sample 1, are significantly lower than 5, which suggest that the samples have parent uranium deficiency.

Table 3.6

a) Equilibrium condition of standards

Standard U-ore	wt. gm.	At 80-100 kev		At 610 kev			Act. ratio
		count/30 min	cpm	count/30 min	cpm	cpm corr.for eff.by X 3	
S_1(0.471%U_3O_8)	6.61625	14963	498.80	980	32.67	98.00	5.09
S_2(0.313%U_3O_8)	5.32849	8952	298.40	636	21.20	63.60	4.71
S_3(0.418%U_3O_8)	6.71155	10216	340.53	632	21.07	63.20	5.38
S_4(0.375%U_3O_8)	5.55980	10655	355.17	728	24.27	72.80	4.87

Table 3.6 b) Equilibrium condition of samples

Sample	Wt.(gm.)	% ThO_2 at 2.61 Mev. for corr.	At 80 - 100 Kev.		At 610 Kev.			Act. ratio
			Act./5 min.	Act./5 min. corr.for ThO_2	Act./5 min.	Act./5 min. corr.for ThO_2	Act./5 min. corr.for eff. x 3	
Mon.NBL 7-A	11.72525	9.7% ThO_2 0.35% U_3O_8	15233	3953.01	2682	258.88	776.64	5.08
ε_1	6.61625	(0.471%U_3O_8)	2494	2494.00	163.33	163.33	489.99	5.08
1	12.86170	6.12	10170	2544.33	1813	174.89	524.67	4.85
T	16.17780	8.99	18860	5662.98	3568	733.07	2199.21	2.58
G_1	16.59370	6.52	13140	3624.57	2688	643.93	1931.79	2.60
G_2	15.11015	6.56	13800	4865.77	2678	758.79	2276.37	2.14
B	13.75410	6.50	12025	3089.05	2213	293.42	880.26	3.51
6.1	12.26555	6.54	12832	3571.28	2422	432.65	1297.95	2.75
7	12.96362	7.80	15627	4104.94	3093	617.88	1853.64	2.21
9	14.31908	6.15	11792	2733.72	2296	350.14	1050.42	2.60
10	15.24835	6.55	13285	3211.06	2782	616.96	1850.88	1.73
11.1	13.86610	6.82	13940	4099.80	2603	489.17	1467.51	2.79
11.2	13.69723	6.72	14344	4705.38	2608	537.47	1612.41	2.92
A	13.17942	7.08	14946	4877.80	2984	821.19	2463.57	1.98

3.1.3 Direct determination of U_3O_8 by γ-activity measurement under the 1.76 Mev peak Detector A was used for this purpose. The parameters of the counter were set similar to the experiment described in section 3.1.1. The thorium contents in monazite samples were corrected using the monazite NBL-7A which 0.35% U_3O_8 content was already corrected using the U-ore S_1. The results of the measurements are presented in Table 3.7.

Table 3.7

U_3O_8 contents in monazite samples from intensity measurement at 1.76 Mev. using detector A, set at gain 2, window width 2
BG = 25 C/30 min.

Sample	Wt.(gm.)	Concentration of ThO_2 from 2.61 Mev. %	Total wt. of ThO_2 in sample gm.	Total obs. act. after bg. subtraction c/30 min.	Total act. cpm.	Act. of thorium in sample cpm.	Act. of uranium after corr. count of ThO_2 cpm.	Total wt. of U_3O_8 in sample gm.	Concentration of U_3O_8 from 1.76 Mev. %
Mon.NBL-7A	4.0497	(9.7)[1]	0.3928	3880	129.3	102.8[2]	26.5[3]	0.0142	(0.35)[1]
S_1	1.8989	-	-	500	16.7	-	16.7	0.0089	(0.471)[1]
1	4.3392	6.12	0.2656	4486	149.5	69.5	80.0	0.0430	0.99
T	5.1121	8.99	0.4596	5036	167.9	120.3	47.6	0.0260	0.51
C_1	5.0824	6.52	0.3314	3757	125.2	86.7	38.5	0.0210	0.41
G_2	4.7428	6.56	0.3111	3691	123.0	81.4	41.6	0.0220	0.46
B	4.7876	6.50	0.3112	3109	103.6	81.5	22.2	0.0120	0.25
6.1	4.9313	6.54	0.3225	3721	124.0	84.4	39.6	0.0210	0.43
7	5.1442	7.80	0.4013	4462	148.7	105.0	43.7	0.0240	0.47
9	5.1292	6.15	0.3155	3479	115.9	82.6	33.4	0.0180	0.35
10	5.3561	6.55	0.3508	4107	136.9	91.8	45.1	0.0240	0.45
11.1	5.0247	6.82	0.3427	3914	130.5	89.7	40.8	0.0220	0.44
11.2	4.9950	6.74	0.3367	3896	129.9	88.1	41.8	0.0230	0.46
A	4.9523	7.08	0.3506	2876	95.9	91.8	4.1	0.0022	0.04

Note : 1. Values in brackets are reference values.
2. Values after correction for the 0.35% U_3O_8 content using the S_1-standard.
3. Calculated value.

3.1.4 Simultaneous determination of the ThO_2 and U_3O_8 contents in monazite sample by γ-activity measurements at 0.61 and 0.91 Mev The intensities under the 0.61 and 0.91 Mev γ-ray peak regions were measured by detector A, which parameter were set as already described in section 3.1.1. In order to evaluate the constants for the U_3O_8(in RE) and the ThO_2, the U-ore S_1 and the monazite NBL-7A which U_3O_8 contributions were corrected, were used. The results obtained are shown in Table 3.8.

Table 3.8

Analysis of samples from intensity measurements at 0.61 and 0.91 Mev peaks

Constants for the solution of simultaneous equations:-

K_{U6} = 1595.52 counts per 10 min./gm per 1% U_3O_8 at 0.61 Mev.
K_{U9} = 388.73 counts per 10 min./gm per 1% U_3O_8 at 0.91 Mev.
K_{Th6} = 365.95 counts per 10 min./gm per 1% ThO_2 at 0.61 Mev.
K_{Th9} = 336.47 counts per 10 min./gm per 1% ThO_2 at 0.91 Mev.

Table 3.8

(continued)

Sample	wt. (gm.)	Act/10 min./gm at 0.61 Mev.	Act/10 min./gm at 0.91 Mev.	% Th		% ThO_2	% U_3O_8
				NAA	from Simultaneous eq.	from Simultaneous eq.	
Mon.NBL-7A	4.0497	3549.7	3263.8	(8.52)	(8.52)	(9.7)	(0.35)
S_1	1.8989	751.5	183.1	-	-	-	(0.47)
Th-ore 79-A	2.4989	512.3	471.1	-	1.26(1.00)	1.44	*(0.04)
1	4.3392	3182.7	2278.5	6.18	5.34	6.08	0.60
T	5.1121	4481.1	3235.9	7.16	7.54	8.67	0.82
G_1	5.0824	2931.1	2177.4	6.26	5.96	6.79	0.12
G_2	4.7428	2306.9	1959.4	5.76	4.92	5.65	0.15
B	4.7876	3113.5	2196.2	5.76	5.88	6.70	0.25
6.1	4.9313	2970.2	2202.6	5.64	5.20	5.98	0.49
7	5.1442	3267.6	2511.2	7.28	6.08	6.93	0.46
9	5.1292	3066.7	2132.4	5.52	5.46	6.28	0.50
10	5.3561	3068.1	2313.4	6.52	5.56	6.33	0.74
11.1	5.0247	2964.9	2547.9	6.56	6.51	7.48	0.08
11.2	4.9950	3486.1	2536.1	7.43	5.92	6.82	0.62
A	4.9523	3701.8	2638.0	6.36	6.11	7.02	0.71

Notes:- 1. Values in brackets indicate reference values for standards.

2. * = not calculable.

The results of the ThO_2 contents obtained by the present method agree within 10% with those obtained by γ-activity measurements at 2.61 Mev. The results of the U_3O_8 contents by the present method are not consistant with those obtained by the γ-activity measurement at 1.76 Mev. This might be a consequence of the non-existing RE of the U-family in the samples.

3.1.5 Determination of U_3O_8 in samples in which U_3O_8 in RE and U_3O_8 out of RE are present together From section 3.1.2.2 it was observed that the intensity ratios at 80 - 100 Kev to that at 610 Kev are very low for the monazite samples while they are comparable for the uranium ores S_1, S_2, S_3 and S_4. This suggests that the uranium family in the monazite samples is out of RE. Attempts were made to use the intensity measurements for computing the percent contents of U_3O_8 which is in RE and which is out of RE. From the results which are presented in Table 3.9, it could be observed that when the sample also contains ThO_2 and the ThO_2 content is much higher than the U_3O_8 content, the U_3O_8 in RE and the U_3O_8 out of RE cannot be calculated from the intensity measurements in the 80 - 100 Kev and 610 Kev regions.

3.2 Determination of the uranium and thorium contents by NAA

Monazite NBL 7-A was used as standard to avoid the difference in neutron flux between the sample and the standard which might occur due to the high concentration of rare earths in the samples. The standard and the samples, weighing 50 mg each, were subjected to irradiations for 2 minutes. The γ-spectra were measured, two minutes each, via detector D, which was previously calibrated to give an energy of 1.4 Kev/channel. The γ-spectrum of the standard NBL-7A is shown in Figure 3.8. The area under the 29.2 Kev peak was used for the evaluation of the contents of thorium. The decay curves of the 29.2 Kev peak from the standard NBL-7A and the sample G_2 are shown in Figure 3.9 . The decay curves agree well with the half-lives of Th^{233}. It is therefore shown that the 29.2 Kev peak can be used selectively for the quantitative determination of thorium in monazite samples, since no interferences from other nuclides were discovered. The results of the analysis are shown in Table 3.10.

Table 3.9

U_3O_8 content in samples in which the uranium family is partly out of RE.

BG.at 80 Kev. = 56 cpm.

at 610 Kev. = 4 cpm.

Sample	wt. (gm.)	80 Kev.		610 Kev.		% ThO_2 for correction	% U_3O_8	
		Net cpm. sub. BG.	sp.act.U_3O_8 gm^{-1} %oxide^{-1} corr.for ThO_2	Net cpm. sub. BG.	sp.act.U_3O_8 gm^{-1} %oxide^{-1} corr.for ThO_2		in RE	out of RE
Synthesized U_3O_8 (out of RE)	3.0593	6115	121.4	89	1.8	-	-	(16.47)
S_1 (in RE)	1.8989	329	367.9	24	26.8	-	(0.471)	-
Mon.NBL 7-A	4.0497	2452	-	267	-	(9.7)	(0.35)	-
1	4.3392	1682	86.8	181	6.1	6.12	0.22	0.11
T	5.1121	3119	168.3	377	21.4	8.99	0.88	-1.28
G_1	5.0824	2107	94.1	270	15.3	6.52	0.65	-1.21
G_2	4.7428	2174	135.9	269	18.5	6.56	0.77	-1.20
B	4.7876	1921	81.8	245	13.3	6.50	0.57	-1.03
6.1	4.9313	2065	97.3	275	17.7	6.54	0.76	-1.37
7	5.1442	2498	102.2	345	21.7	7.80	0.94	-1.95
9	5.1292	1776	43.9	263	15.5	6.15	0.69	-1.73
10	5.3561	2085	67.4	269	12.1	6.55	0.52	-1.01
11.1	5.0247	2247	112.0	272	14.4	6.82	0.60	-0.88
11.2	4.9950	2299	129.9	268	14.5	6.72	0.59	-0.70
A	4.9523	2508	158.5	300	19.4	7.08	0.79	-1.03

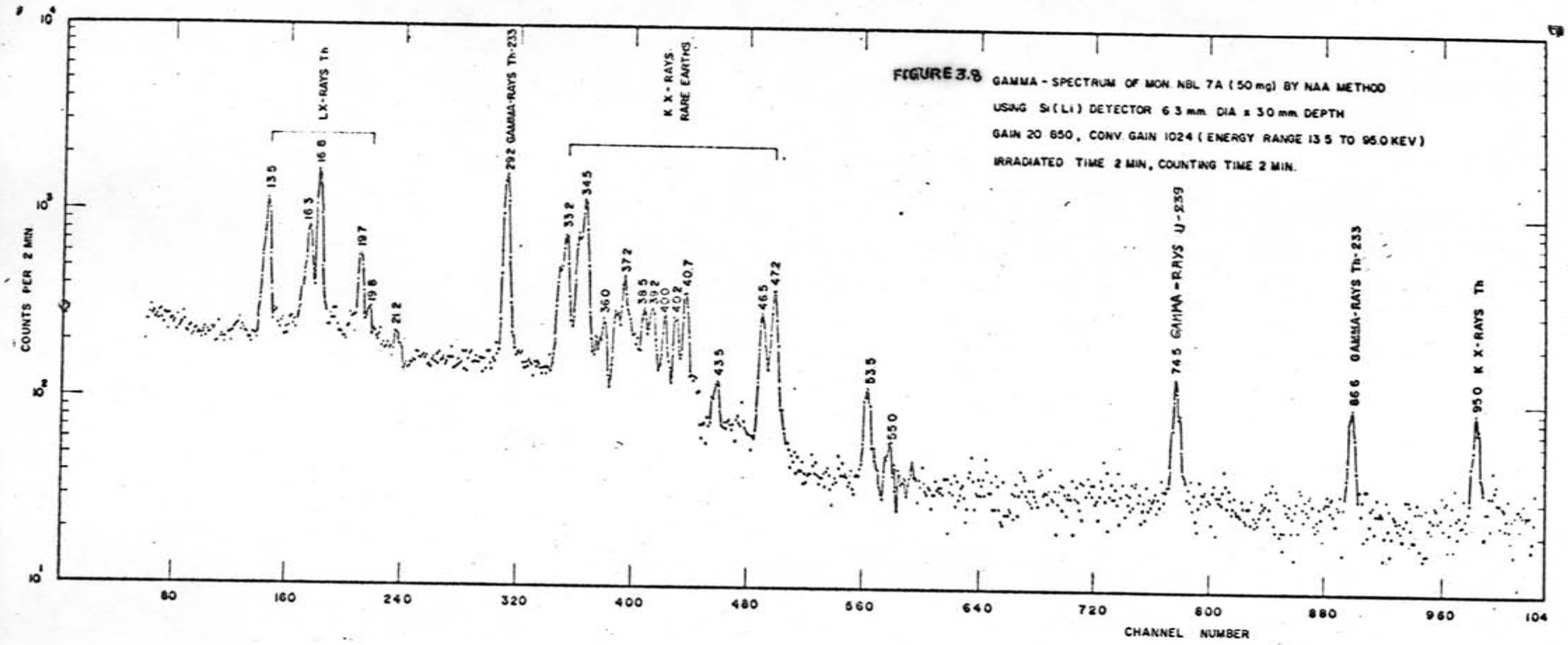


FIGURE 3.8 GAMMA - SPECTRUM OF MON. NBL 7A (50 mg) BY NAA METHOD
USING Si(Li) DETECTOR 6.3 mm DIA x 3.0 mm DEPTH
GAIN 20 650, CONV. GAIN 1024 (ENERGY RANGE 13.5 TO 95.0 KEV)
IRRADIATED TIME 2 MIN, COUNTING TIME 2 MIN.

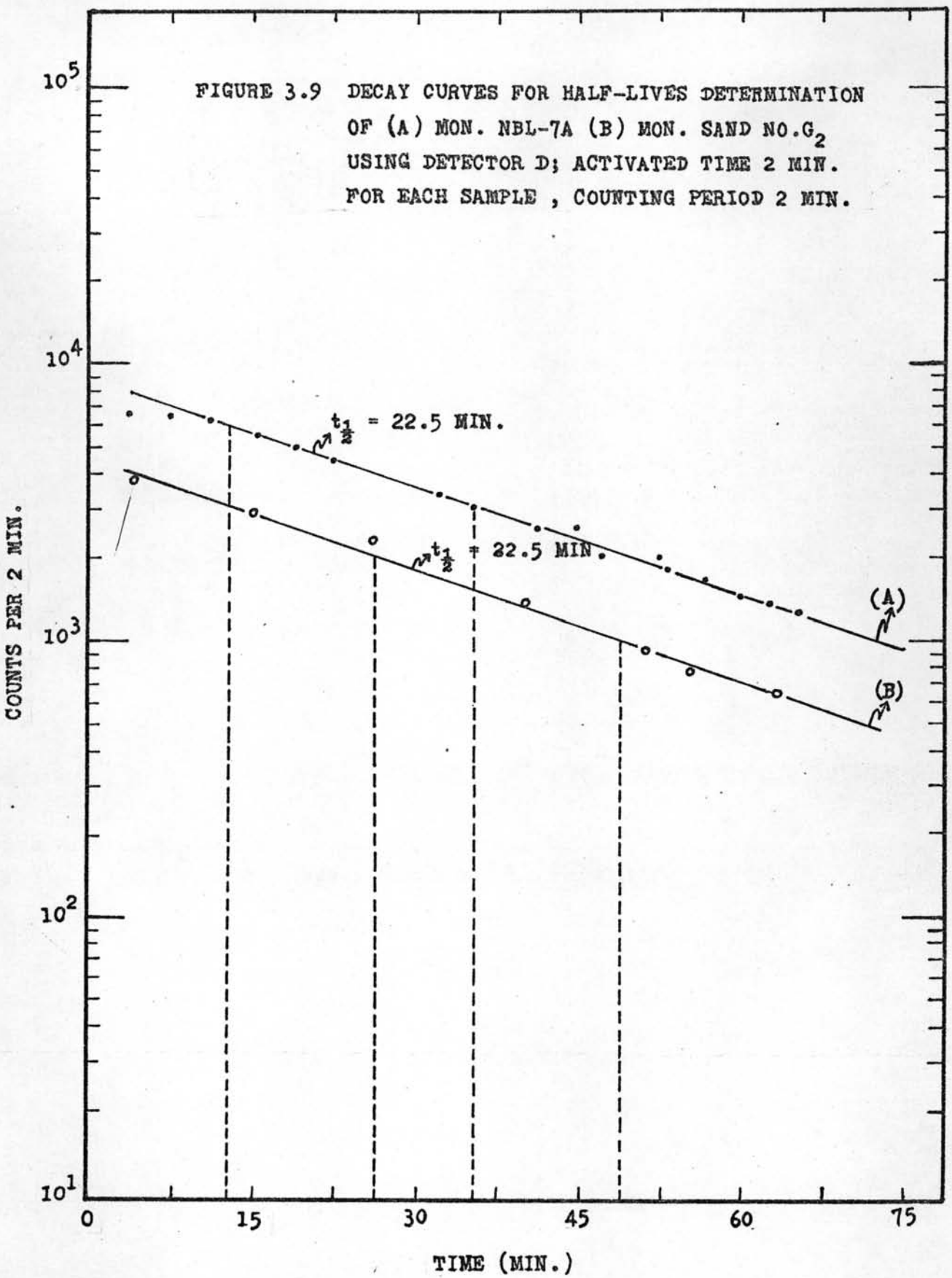


FIGURE 3.9 DECAY CURVES FOR HALF-LIVES DETERMINATION OF (A) MON. NBL-7A (B) MON. SAND NO.G_2 USING DETECTOR D; ACTIVATED TIME 2 MIN. FOR EACH SAMPLE , COUNTING PERIOD 2 MIN.

Table 3.10

Determination of ThO_2 by NAA; weight of sample 50 mg ; activated time 2 min.; counting time 2 min.

Sample	% Th 1st run	% Th 2nd run	% Th 3rd run	average % Th $\bar{x} \pm \sigma$
1	6.01	6.38	6.14	6.18 ± 0.27
T	7.49	7.49	7.85	7.16 ± 0.48
G_1	6.26	6.48	6.04	6.26 ± 0.27
G_2	5.53	5.92	5.82	5.76 ± 0.28
B	5.51	5.41	6.35	5.76 ± 0.45
6.1	-	5.74	5.54	5.64 ± 0.32
7	-	7.09	7.47	7.28 ± 0.44
9	-	5.83	5.21	5.52 ± 0.56
10	-	6.24	6.79	6.52 ± 0.53
11.1	-	6.65	7.11	6.56 ± 0.57
11.2	-	7.10	7.55	7.43 ± 0.48
A	-	6.67	6.04	6.36 ± 0.57

Note :- - = not measured

σ = standard deviation on the mean.

The quantitative analysis of thorium by counting the low-energy γ-peaks after short time irradiation is simple and rapid. The present techniques allow the analysis of 60 samples per man day.

Uranium can also be quantitatively determined by NAA using the 74.5 Kev peak. Since the Si/Li detector was found to have some defects when the analysis of uranium in monazite samples was planned, the experiments were therefore cancelled.

3.3 Spectrophotometric method

The spectrophotometric method described in section 2.3.3 for the determination of the thorium content in monazite samples was checked by analysing the thorium content in the standard monazite NBL-7A. Results of three replicate runs are shown in Table 3.11.

Table 3.11

The thorium content of monazite NBL-7A by spectrophotometric method.

Run No.of solution B	Absorbance at 545 nm.	μg Th/ml in solution B	% Th found in Mon.NBL-7A	Average value of % Th	Chemical yeild %
1	0.384	6.10	7.63		
2	0.383	6.09	7.61	7.62±0.07*	89.4
3	0.384	6.10	7.63		

*Standard deviation on the mean.

The reproducibility of the method is excellent. The results of three runs give a standard deviation of 0.07 . The loss of 10.6% ocurred during digestion and extraction. The processes of digestion took four hours while the process of extraction took only two. Three samples can be analysed in two days per man.

3.4 Titrimetric method

The titrimetric method described in section 2.4.3 for the determination of the thorium content in monazite samples was checked by analysing the thorium content in the standard monazite NBL-7A. A portion of 10 ml from solution B was titrated with 10^{-3}M EDTA. The results of four replicate runs are presented in Table 3.12.

Table 3.12

Thorium content in Mon.NBL-7A by EDTA titration

Solution from Mon.NBL-7A Run No.	ml.buffer solution	10^{-3}EDTA ml.	% Th		% Th Ref.
			obs.value	ave.$\pm\sigma$	
1	8	6.85	7.33		
2	8	6.80	7.23	7.15$\pm$0.24*	8.52
3	8	6.55	6.95		
4	8	6.50	6.91		

*Standard deviation on the mean.

The reproducibility of the method is acceptable. The results of four runs give a standard deviation of 0.24 and a chemical yeild of 83.9%. Since the processes of digestion and extraction are tedious, the method is not recommended for routine work.