

## THE EFFECT OF COUNTING CONDITIONS ON DETERMINING RADIOSTRONTIUM AND OTHER BETA EMITTERS BY LIQUID SCINTILLATION

M. M. RIBERA, J. S. MESTRES, J. F. GARCIA and GEMMA RAURET

Laboratori de Radiologia Ambiental, Departament de Química Analítica, Universitat de Barcelona-Institut d'Estudis Catalans, Diagonal 647, E-08028 Barcelona, Spain

**ABSTRACT.** We report a systematic study of the effects of the most important parameters on the use of the liquid scintillation counters for determining low levels of  $^{90}\text{Sr}$  activity in environmental samples. The parameters studied were: 1) types of scintillator; 2) counting sample composition; 3) counting volume; and 4) counting mixture composition (ratio aqueous solution/scintillation cocktail). For cocktail scintillators, we used Insta-Gel®, Insta-Gel® II and Pico-Fluor™ 40 (Packard). We optimized the Insta-Gel® scintillation cocktail using the Simplex algorithm. We also report a parallel study for  $^{14}\text{C}$  and tested Insta-Gel®, PPO, PPO-dMePOPOP and butyl-PBD. The optimum working conditions for this technique were calculated with figure of merit ( $\text{FM} = E^2/B$ ) and the optimization function, OF, which depends on variables 3, 4 and the working window. Our results showed the great importance of the counting volume and the composition of the counting mixtures for  $^{90}\text{Sr}$  and  $^{14}\text{C}$ . With this study, we were able to establish our counting method.

### INTRODUCTION

The most important beta-emitting radionuclides that should be monitored in the environment are radiostrontium, radiocarbon and tritium.  $^{14}\text{C}$  and  $^3\text{H}$  are low-energy beta-emitting radionuclides ( $\beta$  max. 18.6 keV for  $^3\text{H}$  and 156 keV for  $^{14}\text{C}$ ). Liquid scintillation counting (LSC) is commonly used for their measurement.  $^{89}\text{Sr}$  and  $^{90}\text{Sr}$  are high-energy  $\beta$  emitters (for  $^{89}\text{Sr}$   $\beta$ max. = 1.463 MeV and for  $^{90}\text{Sr}$   $\beta$ max. = 0.546 MeV), the latter being in secular equilibrium with  $^{90}\text{Y}$  ( $\beta$ max. = 2.280 MeV). For this reason, the activity of  $^{90}\text{Sr}$  is measured mainly by proportional gas counting (Haddad & Zikovsky 1988; Ibbett 1967; Kramer & Davies 1982; Talvitie & Demint 1965; Tsubota 1963), a technique recommended by several organizations (Baratta 1984; EPA 1979; HASL 1977; Mingot *et al.* 1987). In some cases,  $^{90}\text{Sr}$  is measured by the Cerenkov effect (Harvey *et al.* 1989; Randolph 1975) or by LSC (Amano & Yanase 1990; Benzi, Operti & Volpe 1988; Horwitz, Dietz & Fisher 1991; Mishra & Parks 1987; Schönhofer & Weisz 1987; Tait, Wiechen & Behrens 1989). The determination of  $^{14}\text{C}$  by LSC in environmental samples is accomplished either by LSC of benzene synthesized with carbon from sample or by LSC of Carbo-Sorb® counting  $\text{CO}_2$  from sample.  $^{90}\text{Sr}$  is determined in solid phase (gas counting) or in aqueous solution (LSC) or by the Cerenkov effect in environmental samples.

The high efficiency of LSC makes this technique particularly suitable for the measurement of very low activity levels. It can be applied to monitoring  $\beta$ -emitting radionuclides in any study of the impact of the nuclear industry, in which the environmental samples have very low activity levels. One of the most important counting parameters is the working window, which is optimized with the figure of merit: maximum value obtained in  $\text{FM} = E^2/B$ , where  $E$  is the efficiency and  $B$  is the background. Another important parameter in low-activity measurements is the total counting volume, which causes an increase in total activity along with an increase in background. However, when low-activity levels are measured in aqueous solution, the water content in the counting mixture is of paramount importance, because water is a quencher. Other parameters to consider are types of scintillation cocktails and vials, and, for  $^{90}\text{Sr}$ , counting sample composition.

Previously we used polyethylene vials to measure  $^{90}\text{Sr}$  and glass vials for  $^{14}\text{C}$  (Rauret, Mestres & Garcia 1989, 1990). Here we report a systematic study of the effect of the type of scintillator, counting sample composition, counting volume and counting mixture composition (ratio aqueous

solution/scintillator). We established the optimum working conditions using two functions: FM and the optimization function, OF (Rauret *et al.* 1990), which depends on counting volume and counting mixture composition. We optimized  $^{90}\text{Sr}$  using the Simplex approach (Deming & Morgan 1973) for Insta-Gel® and Pico-Fluor™ 40 scintillation cocktails.

## METHODS

For  $^{90}\text{Sr}$ , we used a Berthold LB5040 dual LS counter with two counting positions, logarithmic amplification, external standard source of  $^{137}\text{Cs}$  and refrigeration, coupled to a Silena multichannel analyzer with 4096 channels (1–2000 keV range) consisting of a display unit, Model 7940/1, and a basic unit, Model 7934. For  $^{14}\text{C}$ , we used a Tri-Carb® 2000CA/LL with linear amplification, a multichannel analyzer with 4096 channels (1–2000 keV range), a variable-range background discriminator based on the three-dimensional spectrum analyzer, spectralyzer, three independent regions with variable limits for data accumulation, external standard source of  $^{133}\text{Ba}$ , spectrum storage possibility and refrigeration.

We used a standard 20-ml polyethylene vial for  $^{90}\text{Sr}$  and a standard low-potassium borosilicate 20-ml glass vial for  $^{14}\text{C}$  (both from Packard). We also used the following reagents: 1) for  $^{90}\text{Sr}$ : a) solution of labeled  $^{90}\text{Sr}$  in equilibrium with  $^{90}\text{Y}$  (from Amersham International plc) with an activity in  $^{90}\text{Sr}$  of  $2.66 \times 10^5$  dpm  $\text{g}^{-1}$  (15 September 1987). From this standard, a solution, S1, with  $1.39 \times 10^4$  dpm  $\text{g}^{-1}$  was prepared; b)  $\text{Sr}(\text{NO}_3)_2$  and  $\text{HNO}_3$  analytical grade; c) distilled water; d) scintillation cocktails, Insta-Gel® II, Pico-Fluor™ 40 and Insta-Gel® (all from Packard); 2) for  $^{14}\text{C}$ : a) standard toluene labeled with  $^{14}\text{C}$  (Packard) was used to prepare an active solution of 3681 dpm  $\text{g}^{-1}$  in benzene; b) scintillation cocktails: PPO 0.4% (p/v in toluene), PPO 0.4% (p/v) dMe-POPOP 0.01% (p/v in toluene), Bu-PBD 2.5% (p/v in benzene) and Insta-Gel®.

For the preparation and measurement of counting mixtures, we used, for  $^{90}\text{Sr}$ , solutions of different chemical composition prepared from dilution of S1 (Table 1). The concentration of  $\text{Sr}^{2+}$  and  $\text{NO}_3^-$  was obtained by adding  $\text{Sr}(\text{NO}_3)_2$  and  $\text{HNO}_3$ . Different quantities of these solutions were mixed with Insta-Gel® II until the required ratio aqueous solution/scintillation cocktail was achieved. Finally, samples of 8, 12, 16 and 20 ml of the prepared mixture were added and weighed into a previously weighed vial. Background solutions of the required composition were prepared with  $\text{Sr}(\text{NO}_3)_2$ ,  $\text{HNO}_3$  and distilled water in the same way as the active mixtures. Counting solutions for Simplex optimization were prepared by mixing  $\text{Sr}(\text{NO}_3)_2$ ,  $\text{HNO}_3$ , distilled water, solution S1 and scintillation cocktail (Insta-Gel® or Pico-Fluor™ 40) according to the values given by the Simplex algorithm, to a total volume of 20 ml. Background solutions were prepared in the same way. The vials were stored for at least 2 h in the refrigeration compartment before counting, in order to obtain a stable temperature and to avoid any luminescence. Backgrounds were measured for 24–36 h and samples counted for 40–50 min each.

For  $^{14}\text{C}$ , the counting solution for scintillation cocktail studies had a total volume of 3 ml, whereas for volume studies, 3, 5 and 7 ml were used. The ratio of benzene to scintillation cocktail was kept constant at 4:1. The active samples were prepared by weighing 1 ml of active benzene, and adding 1.4 ml of inactive benzene and 0.6 ml of scintillation cocktail for samples with a total volume of 3 ml, and, for the others, by weighing 1 ml of active benzene and adding the corresponding volumes up to 5 or 7 ml of inactive benzene and scintillation cocktails, to maintain the benzene/scintillation cocktail proportions. Inactive benzene was used for background. Counting time was 1 h for active solutions and 6 h for background solutions.

**RESULTS AND DISCUSSION**

For <sup>90</sup>Sr, the first results obtained using Insta-Gel® II showed that the counting volume (V) and the composition of the counting mixture (f = aqueous solution/scintillator) were the most significant variables in the measurement, whereas sample composition seemed unimportant. Tables 2, 3 and 4 show our results, for different f and V values at different compositions. For f values >0.8, the counting solutions separated into two distinct phases. We observed similar behavior for lower f values 48 h after adding scintillation cocktail. The Simplex method was applied at constant volume (V = 20 ml) and for three variables, which range as follows:

- Variable 1: % aqueous phase (% f) = 5–70% (v/v)
- Variable 2: Sr<sup>2+</sup> concentration = 2–25 mg ml<sup>-1</sup>
- Variable 3: NO<sub>3</sub><sup>-</sup> concentration = 0.25–6 mol liter<sup>-1</sup>.

TABLE 1. Concentrations of Sr<sup>2+</sup> and NO<sub>3</sub><sup>-</sup>

Concentrations	S2	S3	S4	S5	S6	S7	S8	S9	S10
Sr <sup>2+</sup> (g liter <sup>-1</sup> )	5	10	5	10	15	1	10	15	5
NO <sub>3</sub> <sup>-</sup> (mol liter <sup>-1</sup> )	0.25	0.25	1	1	1	2	2	2	4

TABLE 2. Results Obtained With [NO<sub>3</sub><sup>-</sup>] = 0.25 mol liter<sup>-1</sup> and [Sr<sup>2+</sup>] = 10 mg ml<sup>-1</sup>

f(v/v) (%)	Parameters	V = 8	V = 12	V = 16	V = 20
8	Window	162–282	151–284	158–284	149–284
	E(%)	77.83	83.26	81.13	83.80
	B(cpm)	8.27	10.37	9.67	11.57
	FM	2928.4	2674.2	2723.6	2428.2
	OF	2.799	1.981	1.444	1.223
12	Window	157–278	152–281	146–281	150–279
	E(%)	79.36	82.73	84.30	81.82
	B(cpm)	8.58	10.35	11.64	10.67
	FM	2936.1	2645.7	2441.0	2509.1
	OF	1.912	1.343	1.046	0.828
25	Window	139–271	131–274	144–284	134–274
	E(%)	81.58	85.57	83.64	84.22
	B(cpm)	9.18	10.82	11.21	11.83
	FM	2901.0	2707.7	2497.3	2399.0
	OF	0.935	0.647	0.504	0.411
35	Window	147–275	133–280	133–280	132–278
	E(%)	78.29	84.80	84.58	84.35
	B(cpm)	9.04	11.57	11.67	11.88
	FM	2711.3	2485.3	2450.9	2394.9
	OF	0.676	0.472	0.358	0.303
50	Window	64–260	79–263	94–264	101–264
	E(%)	94.45	94.07	90.17	88.69
	B(cpm)	14.11	14.31	13.69	13.30
	FM	2529.3	2474.0	2376.4	2366.4
	OF	0.590	0.385	0.305	0.202

TABLE 3. Results Obtained With  $[\text{NO}_3^-] = 1 \text{ mol liter}^{-1}$  and  $[\text{Sr}^{2+}] = 15 \text{ mg ml}^{-1}$ 

f(v/v) (%)	Parameters	V = 8	V = 12	V = 16	V = 20
8	Window	127-287	151-289	157-288	148-287
	E(%)	88.47	82.75	80.90	83.28
	B(cpm)	12.17	10.07	10.36	11.13
	FM	2571.6	2720.2	2527.7	2491.7
	OF	2.911	1.838	1.416	1.147
35	Window	66-267	70-265	79-266	94-269
	E(%)	92.71	90.50	91.22	90.86
	B(cpm)	13.64	14.68	14.78	14.77
	FM	2520.1	2231.3	2251.4	2236.5
	OF	0.636	0.461	0.348	0.267
50	Window	73-262	69-262	74-261	75-256
	E(%)	91.23	92.22	91.04	90.21
	B(cpm)	13.11	13.87	14.14	14.06
	FM	2540.0	2453.2	2343.9	2314.6
	OF	0.408	0.344	0.242	0.187

TABLE 4. Comparison of Different Compositions\* (Volume = 20 ml)

f(v/v) (%)	Parameters	CX	CY	CZ	DX
50	Window	65-251	58-247	63-254	49-222
	E(%)	89.79	92.98	93.53	90.62
	B(cpm)	15.72	14.82	15.59	13.45
	FM	2051.5	2332.5	2244.2	2443.2
	OF	0.187	0.191	0.196	0.181
60	Window	58-240	52-243	51-236	37-209
	E(%)	90.09	92.93	92.07	87.31
	B(cpm)	13.47	15.25	14.87	13.09
	FM	2409.7	2265.9	2279.6	2329.8
	OF	0.159	0.161	0.161	0.153
70	Window	54-228	50-230	26-231	--
	E(%)	89.39	90.98	89.70	--
	B(cpm)	13.00	13.62	13.23	--
	FM	2459.4	2431.4	2432.8	--
	OF	0.134	0.135	0.134	--

\*Compositions: C ( $[\text{NO}_3^-] = 2 \text{ mol liter}^{-1}$  with X = 5, Y = 10 i Z = 15 mg  $\text{Sr}^{2+} \text{ ml}^{-1}$ ) and DX ( $[\text{NO}_3^-] = 4 \text{ mol liter}^{-1}$  with 5 mg  $\text{Sr}^{2+} \text{ ml}^{-1}$ )

Table 5 shows the results obtained with Insta-Gel® scintillation cocktail. The most important variable was  $f$ , which easily achieved a maximum.  $\text{Sr}^{2+}$  and  $\text{NO}_3^-$  concentrations show little effect and many experiments were necessary to reach a maximum. Optimum  $\text{Sr}^{2+}$  and  $\text{NO}_3^-$  concentrations ranged between 13.16 and 16.08  $\text{mg ml}^{-1}$  and between 2.33 and 2.58  $\text{mol liter}^{-1}$ , respectively.

TABLE 5. Real Values of Simplex Variables

Vertex	Var. 1: %f (w/w)	Var. 2: $[\text{Sr}^{2+}]$ ( $\text{mg ml}^{-1}$ )	Var. 3: $[\text{NO}_3^-]$ ( $\text{mol liter}^{-1}$ )	Response (OF)
1	5.058	2.000	0.250	1.933
2	67.584	7.421	1.605	0.142
3	22.432	23.684	1.605	0.438
4	23.942	7.421	5.671	0.409
5P <sub>ref</sub>	71.817	23.684	5.671	--
6P <sub>con</sub>	22.248	7.421	1.605	0.460
7P <sub>ref</sub>	55.139	18.263	4.316	0.176
8P <sub>ref</sub>	74.672	2.000	6.000	--
9P <sub>con</sub>	39.025	17.360	2.735	0.254
10P <sub>ref</sub>	(70%)	21.275	0.250	--
11P <sub>con</sub>	39.691	10.885	4.278	0.247
12P <sub>ref</sub>	64.248	7.020	4.066	--
13P <sub>con</sub>	49.122	14.775	3.067	0.199
14P <sub>ref</sub>	72.690	16.088	1.714	0.131
15P <sub>dil</sub>	72.006	18.689	0.432	0.141
16P <sub>ref</sub>	72.910	13.074	2.023	0.130
17P <sub>dil</sub>	72.646	12.225	1.500	0.134
18P <sub>ref</sub>	71.548	6.125	0.250	--
19P <sub>con</sub>	65.745	15.229	3.048	0.143
20P <sub>ref</sub>	71.836	9.159	0.513	0.149
21P <sub>con</sub>	68.305	13.712	2.414	0.139
22P <sub>ref</sub>	73.332	21.161	2.495	0.126
23P <sub>dil</sub>	73.283	25.000	2.941	0.129
24P <sub>ref</sub>	72.769	19.836	1.740	0.132
25P <sub>ref</sub>	73.008	13.712	2.414	0.117
26P <sub>dil</sub>	73.099	10.649	2.751	0.130
27P <sub>ref</sub>	73.332	15.877	2.907	0.129
28P <sub>ref</sub>	73.634	20.758	3.187	0.130
29P <sub>con</sub>	73.087	14.995	2.314	0.132
30P <sub>ref</sub>	73.315	17.235	2.801	0.128
31P <sub>ref</sub>	73.117	17.462	2.453	0.129
32P <sub>con</sub>	72.969	15.461	2.609	0.127
33P <sub>ref</sub>	72.930	13.836	2.184	0.130
34P <sub>con</sub>	73.152	16.386	2.647	0.130
35P <sub>ref</sub>	72.996	13.774	2.299	0.129
36P <sub>con</sub>	73.345	15.049	2.531	0.127
37P <sub>ref</sub>	73.212	14.199	2.376	0.127

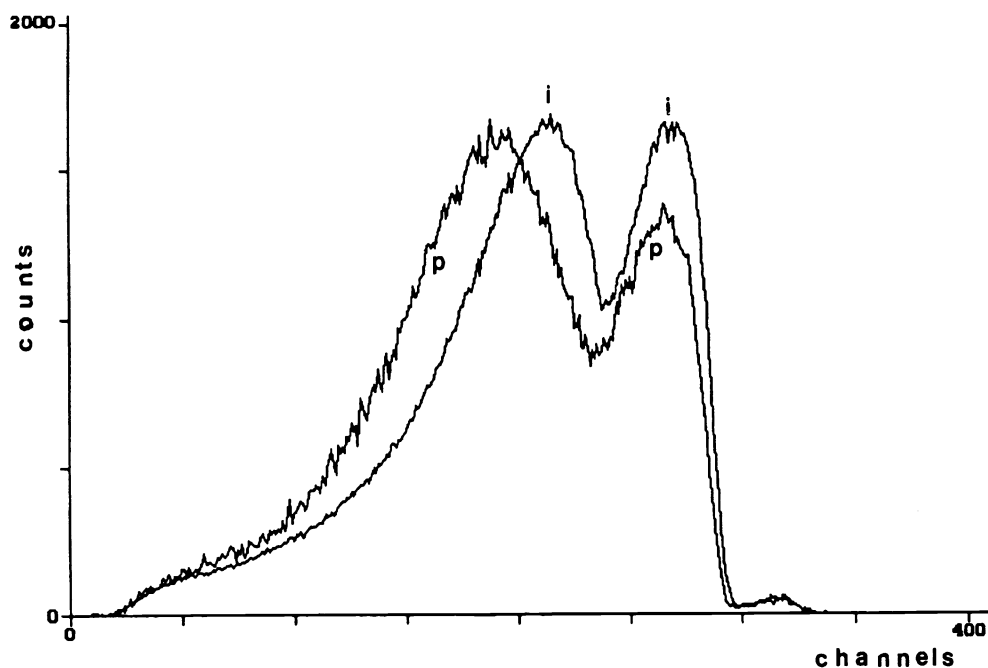


Fig. 1. Comparison between two scintillation cocktails; i = Insta-Gel®; p = Pico-Fluor™ 40

Using Pico-Fluor™ 40, the mixtures prepared according to the values given by Vertex 2, 3 and 4 could not be measured in homogeneous solution. Figure 1 shows the spectra obtained with Pico-Fluor™ 40 and with Insta-Gel® scintillation cocktails in the same conditions (Vertex 1:  $f = 5\%$ ,  $2 \text{ mg ml}^{-1}$  of  $\text{Sr}^{2+}$ ,  $0.25 \text{ mol liter}^{-1}$  of  $\text{NO}_3^-$ ). From the comparison, we conclude that Insta-Gel® is the best scintillation cocktail to measure radiostrontium.

For  $^{14}\text{C}$ , the results obtained for different scintillation cocktails and different total volumes are shown in Tables 6 and 7. The value of  $f$  in the OF is always 0.8 (ratio benzene/scintillation cocktail). Table 6 shows the same behavior for both functions, FM and OF. The different scintillation cocktails produce only slight differences in the results. The most appropriate scintillation cocktail is Insta-Gel®, although according to the peculiarities of the benzene synthesis and the possibility of introducing  $^3\text{H}$  to the benzene molecule during this process, it seemed reasonable to move the lower threshold up to energy values of 16–17 keV. In this situation, the relative performance of scintillation cocktails changes; butyl-PBD is the best (Table 6).

The influence of the total counting volume is illustrated in Table 7, where FM and OF values diverge. Because OF takes into account more variables than FM, this function is considered the most appropriate with optimization processes. Thus, the optimum total volume proposed is the maximum studied, 7 ml, corresponding to 5.6 ml of synthesis benzene and 1.4 ml of scintillation cocktail. This value is approximately the amount generally available with the synthesis devices commonly used in  $^{14}\text{C}$  laboratories.

TABLE 6.  $^{14}\text{C}$  Results Obtained for Different Scintillation Cocktails\*

Scintillation cocktail	Window (keV)	Efficiency (%)	Background (cpm)	FM	OF
Insta-Gel®	12 – 53	52.08	0.643	4216	0.731
Insta-Gel®	16 – 53	43.61	0.487	3908	0.759
Insta-Gel®	17 – 53	41.57	0.480	3601	0.791
PPO	10 – 48	54.93	0.887	3403	0.814
PPO + dMePOPOP	10 – 48	55.31	0.867	3530	0.799
Butyl-PBD	14 – 82	52.39	0.730	3760	0.774
Butyl-PBD	16 – 82	49.64	0.657	3753	0.775
Butyl-PBD	17 – 82	48.27	0.630	3698	0.780

\*Total volume = 3 ml

TABLE 7.  $^{14}\text{C}$  Results Obtained for Different Total Volumes\*

Volume (ml)	Window (keV)	Efficiency (%)	Background (cpm)	FM	OF
3	14 – 82	52.39	0.730	3760	0.774
5	13 – 78	52.60	0.873	3168	0.505
7	13 – 75	51.25	1.213	2165	0.437

\*Scintillation cocktail: Bu-PBD

## CONCLUSIONS

From our results, we conclude that, for  $^{90}\text{Sr}$  measurements by LSC in aqueous phase, the most significant variables are the counting volume (V) and the composition of the counting mixture (f); the type of scintillator is less significant. Sample composition ( $[\text{Sr}^{2+}]$  and  $[\text{NO}_3^-]$ ) given by the separation method used appears to have no influence. The optimum conditions for measuring  $^{90}\text{Sr}$  in aqueous phase are:

Scintillation cocktail: Insta-Gel®  
 Counting volume: 20 ml (polyethylene vial)  
 Ratio aqueous solution/scintillation cocktail: 70% (v/v)  
 $[\text{Sr}^{2+}]$ : 13.16–16.08 mg ml<sup>-1</sup>  
 $[\text{NO}_3^-]$ : 2.33–2.58 mol liter<sup>-1</sup>.

For  $^{14}\text{C}$ , the most significant factor is the total mass of the counting mixture, which produces a variation in the OF values much larger than those obtained when different scintillation cocktails are used. The optimum conditions proposed for the determination of low-level  $^{14}\text{C}$  by LS with glass vials and a ratio benzene/scintillation cocktail 4:1 are:

Scintillation cocktail: Butyl-PBD (2.5% p/v in benzene)  
 Total counting volume: 7 ml (5.6 ml synthesis benzene + 1.4 ml scintillation cocktail).

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