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几种核素放射性标准溶液的研制

DEVELOPMENT OF STANDARD SOLUTION FOR SOME RADIONUCLIDES

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杨景霞：中国原子能科学研究院副研究员，1962年毕业于清华大学工程化学系。

Yang Jir.gxia; Associate professor of China Institute of Atomic Energy. Graduated from Department of Engineering Chemistry, Tsinghua University in 1962.

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几种核素放射性标准溶液的研制

杨景霞　喻以光　陈细林

（中国原子能科学研究院，北京）

摘　要

放射性标准溶液属标准物质类，它是一种重要的标准器具，可用于量值和比对工作中。文中介绍了按制备放射性标准溶液的要求研制的^{60}Co，^{90}Sr-^{90}Y，^{137}Cs，^{134}Cs，^{63}Ni，^{241}Am，^{125}I，^{131}I等8个核素的标准溶液。选择溶液的介质条件为20～50 μg稳定载体/g溶液，酸度为0.1 mol/L的盐酸。碘的放射性溶液调成碱性并加入适当量的稳定剂。溶液经过长期的考察是均匀和稳定的，量值准确可靠。给出的上述核素标准溶液不确定度分别为：0.10%，0.27%，0.18%，0.12%，0.24%，0.18%，0.20%，0.22%（1σ）。

DEVELOPMENT OF STANDARD SOLUTION FOR SOME RADIONUCLIDES

Yang Jingxia Yu Yiguang Chen Xilin
(CHINA INSTITUTE OF ATOMIC ENERGY, BEIJING)

ABSTRACT

Radioactive standard solution is a standard reference material. It is an important standard measuring instrument and used for transferring and intercomparing of quantity value. According to requirement for the preparation of radioactive standard solution, ^{60}Co, ^{90}Sr-^{90}Y, ^{137}Cs, ^{134}Cs, ^{63}Ni, ^{241}Am, ^{125}I, ^{131}I standard solutions have been developed. The medium includes about 20~50 μg stable carrier per gram solution and 0.1 mol/L HCl. For iodine radioactive solutions, the medium is alkaline and the stabilizer is added into it. The solutions are all stable and uniform in the long term. The specific activities are accurate and the uncertainties are 0.10%, 0.27%, 0.18%, 0.12%, 0.24%, 0.18%, 0.20%, 0.22% respectively (1 σ).

INTRODUCTION

Radioactive standard solution and source are important standard reference materials in the field of ionizing radiation metrology. They are used in the transferring and intercomparing of the quantity value of radionuclides. Besides the standard apparatus for measuring activity have been set up, the standard solution and source have been developed to be used for quantity value transferring and intercomparing in many ionizing radiation laboratories in the world. In the present the radiactive solutions and sources produced are various[1,2]. Some of them are in aqueous system and some in organic system.

Because radioactive standard solution is as a kind of standard reference material, its system must be as stable as possible and the specific activity value must be accurate. In many laboratories in the world, a lot of work has been done for preparation, seal and store of standard solutions. IAEA and related organizations have organized such international symposiums[3~6] for improving accuracy and stability of them.

The radioactive solution and source are paid more and more attentions in the radioactive measurement at home. Their applications are becoming wider and wider. But only ten odd kinds of radionuclide standard solutions are produced, it is neccessary to develope more kinds of them.

The following aspects must be met for radioactive standard solution:

(1) The radionuclide has a high purity, i. e. the radioactive impurity must be less than 0.1%.

(2) The solution must be uniform and stable and won't be chemically changed during storing and using.

(3) Radionuclide quantity adsorbed on container wall should be very low and can be ignored.

(4) When evaperated, radioactive substance should not be lost.

(5) It is suitable for further processing such as dilution.

(6) The value of the activity measured (specific activity of the solution) is accurate and reliable.

The procedure of preparation for radioactive standard solution is:

(1) Purity identification

(2) Preparation of radioactive solution

(3) Measuring specific activity

(4) Separate loading, fire sealing and storing

(5) Examination of stability

In this paper, the radionuclides of developed solutions are ^{60}Co, ^{90}Sr-^{90}Y, ^{137}Cs, ^{134}Cs, ^{63}Ni, ^{241}Am, ^{125}I and ^{131}I. Two kinds of specific activity for every one are available: 3.7×10^{5} Bq in volume of about 1 ml and $3.7\times10^{6}\sim10^{7}$ Bq in mass of 3.6 ± 0.2 g.

1 PREPARATION AND MEASUREMENT

1.1 Purity identification

Before a radioactive solution is prepared, it is necessary to separate, purify and identify.

Normally, γ-spectroscope is used to identify its purity for a radioactive solution. No any impurity is found in ^{90}Sr-^{90}Y, ^{63}Ni. The content of impurities for ^{60}Co, ^{137}Cs, ^{134}Cs, ^{241}Am, ^{125}I, ^{131}I is less than 0.1%.

1.2 Preparation

The stable isotope carrier solution is quantitively added into the corresponding radioactive solution. After fully mixed, it is stood for some time (If necessary, heated) so that the isotopes are fully exchanged. An adequate acid and water are used to dilute the solution into a fixed volume in order to obtain expected specific activity of the radioactive solution.

In the preparation process, besides the high purity water and chemical agents are used, a suitable medium is selected (i. e. a certain acidity and carrier), so that container wall adsorption, precipitation and colloid can be avoided. In this way, the solution can be kept uniformly and stably. Generally, the carrier content selected is from 20 to 200 μg/g solution and the acidity is from 0.1 to 0.5 mol/L HCl or HNO_3.

The medium selected in this work is shown in Table 1.

Table 1 Conditions of medium solution

Nuclide	Carrier	Acidity
^{60}Co	~40 μg Co^{2+}/g	0.1 mol/L HCl
^{90}Sr-^{90}Y	~(25 μg Sr^{2+} + 25 μg Y^{3+})/g	0.1 mol/L HCl
^{137}Cs	20 μg Cs^{+}/g	0.1 mol/L HCl
^{134}Cs	20 μg Cs^{+}/g	0.1 mol/L HCl
^{63}Ni	60 μg Ni^{2+}/g	0.1 mol/L HCl
^{241}Am	20 μg La^{3+}/g	0.5 mol/L HCl
^{125}I	50 μg I^{-} + 50 μg $Na_2S_2O_3$ + 5×10^{-4} mol/L NaOH	
^{131}I	50 μg I^{-} + 50 μg $Na_2S_2O_3$ + 5×10^{-4} mol/L NaOH	

Because a negative ion of iodine exists in the solution, the solution is prepared into an alkaline medium and a certain quantity of stabilizer is added into the solution to avoid negative ion of iodine being oxidized into iodine.

Experiments of adsorption by container wall are carried out with different contents of stable carrier. The procedure is as the following:

Arbitrarily take an ampoule of solution to observe whether there is precipitant; then centrifuge it; open the ampoule, draw out the solution completely; centrifuge again, draw out the remainder in the bottom. Wash the ampoule bottom with a little diluted HCl, draw out the washed solution and discharge it; Wash two times again with a little diluted HCl, and once again with a little diluted oxalic acid, draw the washed solutions out and place it into a measuring dish; dry it; measure the radioactivity with a FJ-267 α, β scintillation counter whose efficiency has been calibrated.

Then calculate the percentage of wall adsorption.

The results are listed in Table 2.

Table 2 Influence of carrier content on glass adsorption of ^{90}Sr-^{90}Y

Number	Carrier content (μg/ml)	Adsorption (%)
1	0.0	3.7
2	1.0	0.15
3	5.0	0.069
4	10	0.040
5	20	0.050
6	50	0.045
7	100	0.047

Note: (1) Total activity in original ampoule is 3.7×10^5 Bq, it was measured after the solution has been stored in a month.

(2) data listed in the table are the mean of three samples.

It is shown from Table 2, the bigger the quantity of carrier added is, the lower the adsorption loss is. But when the quantity of carrier is added to a certain amount, the adsorption loss is not changed.

1.3 Measurement of activity

This step is the key for the preparation of radioactive standard solution. Specific activity is measured with certificated 4 πβ-γ coincidence counter or other absolute measuring instruments.

The measurement is divided into three steps: Sampling, source preparing and measuring.

The solution is drawn into a polyethlene ampoule, the neck of which is pulled into a capillary. The mass of the solution dropped onto metallized VYNS film is weighed by the balance (TG332A) which is periodically calibrated.

The step for preparation of source is as the following:

The radioactive solution is dropped onto a VYNS film which is mounted on an aluminium ring (ϕ32 mm). In order to reduce self-absorption of source, silicon-gelethanol sespending solution is electrosprayed on the VYNS film to form a fixed pad (ϕ6~8 mm) on which radioactive solution is dropped. After being dried, it is measured.

The radioactivity of ^{60}Co, ^{241}Am, ^{131}I are directly measured to calculate the specific activity. The efficiency tracing technique is used for measuring the activity of ^{90}Sr-^{90}Y, ^{137}Cs, ^{63}Ni. The efficiency tracer used is ^{60}Co standard solution. Besides ^{60}Co solution, ^{134}Cs standard solution is used as tracer for ^{137}Cs solution too. The extrapolation is used for ^{134}Cs. A well NaI scintillation equipment is used to measure ^{125}I radioactivity and intercompared nationally (Twelve labs altogather).

1.4 Sealing and stocking of the solution

Radioactive solution is usually stored in glass container. When stored after a long period, glass will be dissolved in acid solution [7], whose concentration can reach 0.2 mg/ml in some severe cases [8], deteriorating the solution quality, increasing solid weight on the circular pad of thin film source and effecting the source quality.

Polyethylene vial is not easily broken and has small adsorption of inorganic ions. But its wall has the phenomenon of leakage, it does not suit for storing radioactive solution for long time [9].

Radioactive solution is filled in ampoule (95 type glass) in fuming cupboard, then the ampoule is sealed with fire as quickly as possible and stored in shady, cool and well ventilated place.

In order to check whether there is loss of evaperation during the process, the empty ampoules and the ampoules with nonradioactive solution which have the same compositions as radioactive solution are weighed before and after flame sealing. Evaperating rate of solution from open ampoule is also measured by means of weighing. Results are shown in Table 3 and 4.

Table 3 Ampoule weight before and after flame sealing

Number	weight before flame sealing (g)	weight after flame sealing (g)	weight difference (μg)
1	5.314840	5.314855	+15
2	5.292920	5.292910	−10
3	5.591400	5.591390	−10
4	5.414620	5.414610	−10
5	5.191670	5.191650	−20
6	5.426770	5.426790	+20
7	4.239600	4.239590	−10
8	4.230220	4.230200	−20
9	4.353460	4.353440	−20
10	4.100170	4.100180	+10

Note: Ampoules numbered from 1 to 6 are filled with solution, ampoules numbered from 7 to 10 are empty.

As shown in Table 3, the change of weight of the empty ampoules and the ampoules with solution are within balance error range during the process, so the loss of evaperation can be ignored.

In Table 4, evaperating loss rate of solution in open ampoule is (0.52 ± 0.11) μg/min. It lasts just several minutes from solution being filled into ampoule to the ampoule being sealed with flame, for example: If the time is 5 min, the evaperation loss is just 3 μg or 3×10^{-4}% of 1 g solution, thus the activity change caused by it can be ignored.

Table 4 Evaperation loss rate of solution in ampoule

Number	Time interval (min)	180	180	180	450	900	960	1200
1	Weight loss (μg)	80	78	72	170	302	442	
	Loss rate (μg/min)	0.44	0.43	0.40	0.38	0.34	0.46	
2	Weight loss (μg)	88	112	92	233	432	538	
	Loss rate (μg/min)	0.49	0.62	0.51	0.52	0.48	0.56	
3	Weight loss (μg)	76	89	82	370	445	530	
	Loss rate (μg/min)	0.42	0.49	0.46	0.82	0.49	0.55	
4	Weight loss (μg)	110	110	102		610	588	688
	Loss rate (μg/min)	0.61	0.61	0.57		0.68	0.61	0.57

Average loss rate: 0.52±0.11 (μg/min)

1.5 Stability checking of solution

One of the important characteristics of radioactive solution is that the solution is stable during the process of using and storing. In order to check its stability, three kinds of experiments are carried out: Periodically checking container wall adsorption of radionuclides, checking evaperation and leakage during solution storing and checking specific radioactivity.

1.5.1 Container wall adsorption of radionuclides

Periodically measure container wall adsorptive percentage of radioactive solution after drawing it out of ampoule as mentioned in section 1.2 (relation between adsorption and carrier). At last, the ampoule is broken into pieces and put them into measuring dish to determine their radioactivity.

Experiment results are given in Table 5. The counts are roughly near the background for the washed broken ampoule, except that ^{90}Sr-^{90}Y is several counts higher, whose adsorptive percentage calculated is only $5.2\times10^{-5}\%$.

As seen from Table 5, the wall adsorption to radionuclides in solution with 50 μg carrier ions/ml and 0.1 mol/L acidity is by the level of 10^{-4} which agrees with Ref. [10].

Table 5 Wall adsorption loss of radioactivity

Nuclide	Stored time	Adorption percentage($\times10^{-3}\%$)			
		1st washed HCl solution	2nd washed HCl solution	Oxalic washed solution	total adsorption
^{60}Co	11 days	3.7	2.4	1.1	7.2
	1 month	3.8	2.1	1.0	6.9
	2 month	4.8	3.0	1.8	9.6
	4 month	4.2	2.6	1.4	8.2
	6 month	4.8	3.2	1.3	9.3
	12 month	5.2	3.4	1.2	9.8
	13 month	4.2	3.3	1.4	8.9
	24 month	2.9	2.9	1.2	9.1
^{90}Sr-^{90}Y	11 days	3.7	2.5	1.7	7.9
	1 month	4.0	2.0	4.3	10
	2 month	4.2	2.1	5.0	11
	4 month	5.0	2.4	5.7	13
	6 month	4.8	2.6	6.0	13
	12 month	5.9	3.0	6.8	16
	13 month	5.5	2.5	6.0	14
	24 month	4.8	2.1	6.5	13

Note: Total activity of original solution is 3.7×10^{5} Bq.

1.5.2 Loss weight checking of ampoule

Weigh sealed ampoules with radioactive solution at the same balances regularly to observe whether there are leakage and evaperation of the solution. The weighing result is listed in Table 6.

In Table 6, the weight ranges in scores of micorgrams in two years and does not deviate at the same direction, this difference may be caused by temperature, humidity or the balances operating. Its weight difference percentage is neglegible to one gram of solution. So there is no occurrence of loss weight during solution storing, it is also proved that there are no obvious leakage and evaperation of the solution in this process.

Table 6 Relations between solution weight and stored time

Number	Original weight (g)	Stored time (month)	Present weight (g)	Weight changed (μg)
1	5.827070	1	5.827100	+30
		3	5.827090	+20
		6	5.827040	−30
		12	5.827060	−10
		24	5.827100	+30
2	6.425872	1	6.425835	−37
		3	6.425896	+18
		6	6.425885	+13
		12	6.425860	−12
		24	6.425840	−32
3	5.927860	1	5.927900	+40
		3	5.927840	−20
		6	5.927890	+30
		12	5.927900	+40
		24	5.927847	−13
4	5.32800	1	5.328030	+30
		3	5.328042	+42
		6	5.327980	−20
		12	5.328046	+46
		24	5.328010	+10
5	5.192539	1	5.192548	+9
		3	5.192560	+21
		6	5.192520	−19
		12	5.192534	−5
		24	5.192580	+41
6	5.447500	1	5.447456	−44
		3	5.447510	+10
		6	5.447530	+30
		12	5.447480	−20
		24	5.447475	−25

1.5.3 Specific activity regular checking

Determine specific activity of the solutions in different time intervals to observe whether the solutions are stable during its stocking. The results of all the radionuclides are listed in Table 7～14.

Table 7 Specific activity of ^{60}Co solution

Measured date	Specific activity (Bq/mg)	Normalized specific activity (Bq/mg) 1st batch solution (Refered date: 1988. 3. 2)	2nd batch solution (Refered date: 1991. 11. 22)
24. Nov. 1984	435.68	283.56	
06. Dec. 1984	434.36	283.93	
13. Dec. 1985	379.06	283.39	
13. Apr. 1987	319.50	284.42	
13. Apr. 1987	318.90	283.89	
21. Oct. 1987	296.49	262.83	
03. Dec. 1987	293.24	284.09	
20. Jan. 1988	287.45	283.24	
05. Feb. 1988	285.57	283.01	
02. Mar. 1988	283.27	283.27	
07. Nov. 1989	225.48	281.35	
16. May. 1990	210.88	281.76	
		Average: 283.23±0.91(0.32%)	
22. Nov. 1991	510.05		512.05
16. Dec. 1991	507.05		511.73
17. Dec. 1991	506.87		511.45
20. Feb. 1992	495.17		511.47
			Average: 511.67±0.28(0.05%)

Table 8 Specific activity of ^{90}Sr-^{90}Y solution

Measured date	Specific activity (Bq/mg)	Specific activity normalized to the date 02. March. 1988. (Bq/mg)
24. Nov. 1984	539.20	498.30
05. Dec. 1984	538.66	498.16
13. Dec. 1985	526.93	499.48
14. Jan. 1985	521.26	495.16
15. Apr. 1987	511.50	500.76
02. Mar. 1988	503.68	503.68
03. Mar. 1988	504.37	504.40
		Average: 499.99±3.25(0.65%)

Table 9 Specific activity of ^{131}I solution

Measured date	Specific activity normalized to the time AM. 12:00. 20. March. 1991. (Bq/mg)
AM. 10:00. 02. Mar. 1991	569. 29
20. Mar. 1991	569. 64
02. Apr. 1991	571. 33
09. Apr. 1991	571. 11
mean value	570. 37±0. 99

Table 10 Specific activity of ^{125}I solution

Measured date	Specific activity (Bq/mg)	Specific activity normalized to the time AM. 00:00. 01. May. 1990. (Bq/mg)
PM. 02:00. 16. May. 1990	166. 166	200. 70
29. May. 1990	143. 143	200. 82
mean value measured by twelve labs at home:		200. 76

Table 11 Specific activity of ^{137}Cs solution

Measured date	Specific activity (Bq/mg)	Specific activity normalized to reference time 01. March. 1988. (Bq/mg)
05. Dec. 1987	423. 16	421. 12
21. Jan. 1988	424. 57	423. 51
10. Feb. 1988	425. 17	424. 64
01. Mar. 1988	420. 65	420. 65
24. Jun. 1988	421. 58	424. 80
20. Mar. 1989	411. 33	421. 30
(The upper are efficiency traced by ^{60}Co)		
(The lower are efficiency traced by ^{134}Cs)		
10. Mar. 1989	414. 76	424. 66
23. Mar. 1989	414. 04	424. 26
		Average: 423. 12±1. 79

Table 12 Specific activity of ^{134}Cs solution

Measured date	Specific activity (Bq/mg)	Specific activity normalized to reference time 27. Apr. 1988. (Bq/mg)
20. Apr. 1988	707. 12	702. 55
27. Apr. 1988	705. 56	705. 56
28. Apr. 1988	703. 93	704. 58
10. May. 1988	695. 58	703. 94
24. May. 1988	689. 79	707. 12
26. May. 1988	685. 37	703. 89
28. Jun. 1988	667. 08	706. 19
09. Mar. 1989	529. 92	708. 49
11. Mar. 1989	528. 24	707. 54
23. Mar. 1989	519. 26	703. 24
13. Nov. 1989	516. 11	699. 39
		Average: 704. 77±2. 59(0. 37%)

Table 13 Specific activity of ^{241}Am solution

Measured date	Specific activity (Bq/mg)
04. Jun. 1991	380.24
19. Jun. 1992	381.85
25. Jun. 1992	381.87

Average: 381.32 ±0.94 (0.25%)

Table 14 Specific activity of ^{63}Ni solution

Measured date	Specific activity (Bq/mg)
10. Nov. 1989	356.7
23. May. 1990	353.43
	(The upper are measured by 4πβ-γ coincidence assembly)
(The lower are measured by liquid scintillation assembly)	
27. Jun. 1990	359.50
	Average: 356.56±3.05(0.86%)

As seen from Table 7 to 14, the specific activity corrected by half life is well in agreement for ^{60}Co, ^{90}Sr-^{90}Y solutions in more than two years and for ^{137}Cs, ^{134}Cs solutions in one and half years. Although the data for ^{63}Ni, ^{241}Am solutions are few, their specific activity values change very little from the data acquired in half year. Half lives of radionuclides ^{131}I, ^{125}I are relatively short, the data measured in short term agree well with each other. The national intercomparisons of specific radioactivity of the two radionuclides have been carried out. The data measured by different labs deviats not very much from each other. The values measured in the authors' lab. are comparatively close to the mean. In a word, every radioactive solution developed is uniform, stable with reliable accurate value.

2 ERROR ANALYSIS

For 4πβ-γ coincidence assembly, the main error sources are as follows:

Type A: Statistic fluctuation of counts;

Type B:

a) weighing;

b) correction of dead time;

c) correction of resolving time;

d) background correcting;

If extrapolation method is used, it should be considered:

e) correcting error created by extrapolation;

If efficiency tracing method is used, it also should be considered:

f) correcting error created by determining specific activity of the tracing radionuclide.

Error sources and uncertainties of specific activity of each radionuclide standard solution are listed in Table 15.

Table 15 Various error sources and uncertainties

error types	^{60}Co	^{90}Sr-^{90}Y	^{137}Cs	^{134}Cs	^{125}I	^{131}I	^{241}Am	^{63}Ni
Type A:	0.09	0.25	0.14	0.11	0.20	0.21	0.18	0.21
Type B:								
Dead time	0.01	0.01	0.C	0.01	0.01	0.01	0.01	0.01
Resolving time	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
background	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
weighing	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03
Extropolating		0.032	0.032	0.032		0.05 (Fitting method)		0.10
Nuclide rtacer		0.10	0.10					0.10
$\sqrt{\sum B^2}$	0.035	0.11	0.11	0.047	0.035	0.06	0.035	0.11
$\sqrt{A^2+B^2}$	0.10	0.27	0.18	0.12	0.20	0.22	0.18	0.24

3 CONCLUSIONS

(1) Conditions in preparing radioactive standard solution determined by experiment are: usually 20~200 μg carrier ion/gram (or ml) and 0.1 mol/L HCl or HNO_3 aqueous solution.

(2) Selecting rigid glass as storing container whose radionuclide adsorptive percentage is on the level of 10^{-2}.

(3) For one gram of solution, evaperation loss is very small and inferior to 3×10^{-6} during solution separate loading and flame sealing , in fact, it has no influence on specific activity of solution and can be ignored.

(4) Under the conditions of this experiment, there is no phenomenon occurrence of precipitation, coacervation and coagulation over two years, long-term specific activity values agree with each other within permission of experiment errors, so these standard solutions can be thought as uniform and stable.

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