LSC in Practice

Sample Preparation of ^{36}Cl for LSC

Problem

A researcher was trying to analyze gaseous ^{36}Cl by reaction with a solution of silver nitrate, which forms $Ag^{36}Cl$ precipitate. The $Ag^{36}Cl$ was then dissolved in 2 to 4 N ammonia and 2 mL was added to 15 mL ULTIMA Gold™ (PerkinElmer part number 6013329). This produced a black cloudy solution, which the researcher concluded was not suitable for counting.

The researcher was counting his samples in glass vials at ambient temperature in a PerkinElmer Tri-Carb® 2200CA liquid scintillation counter.

Discussion

The problem lies with the choice of cocktail, in that any cocktail which contains neutralized phosphate esters will show this phenomenon. The black cloudy solution is a precipitate of silver oxide or silver phosphate caused by the presence of phosphate esters. To confirm this, we prepared a test solution as follows:

1. 0.5 mL 10% silver nitrate solution +0.1 mL 1 M nitric acid +0.5 mL 0.15 M sodium chloride.
   This produced a precipitate of silver chloride.
2. To this was added 6.0 mL of 4 N ammonia and the precipitate dissolved.
   This was used as the test solution.
3. 1.0 mL test solution was added to 9.0 mL of various cocktails.

The results are shown in Table 1.

Cocktail	Phosphate Esters	Appearance
ULTIMA Gold	Present	Black cloudy solution
ULTIMA Gold XR	Present	Black cloudy solution
Hionic-Fluor™	Present	Black cloudy solution
Insta-Gel®	None	Clear solution
Pico-Fluor™ 15	None	Clear solution
ULTIMA Gold AB	Trace (< 0.5%)	Clear solution
ULTIMA Gold LLT	Trace (< 0.5%)	Clear solution

Table 1. Suitability of cocktails with $Ag^{36}Cl$/ammonia solution

In addition, 1.0 mL of both 2 N and 4 N ammonia were added to 10 mL ULTIMA Gold, but this mixture remained clear, thus confirming that the problem is not due to the ammonia.

These four cocktails were then checked with the test sample for chemiluminescence (1.0 mL test solution added to 9.0 mL of these cocktails) using a Tri-Carb® 1900 at 19 °C and glass vials. Window settings were as indicated in Table 2.

Cocktail	Window	2 minutes	4 minutes	10 minutes	20 minutes
Insta-Gel (part number 6013399)	0-156 keV	52 CPM	28 CPM	28 CPM	29 CPM
	4-156 keV 0-1,000 keV	21 CPM 61 CPM	16 CPM 37 CPM	19 CPM 34 CPM	21 CPM 37 CPM
Pico-Fluor 15 (part number 6013059)	0-156 keV	54 CPM	37 CPM	26 CPM	29 CPM
	4-156 keV	25 CPM	23 CPM	21 CPM	21 CPM
	0-1,000 keV	60 CPM	43 CPM	42 CPM	40 CPM
ULTIMA Gold AB (part number 6013309)	0-156 keV	55 CPM	43 CPM	27 CPM	28 CPM
	4-156 keV	28 CPM	26 CPM	20 CPM	20 CPM
	0-1,000 keV	60 CPM	50 CPM	34 CPM	35 CPM
ULTIMA Gold LLT (part number 6013377)	0-156 keV	53 CPM	40 CPM	25 CPM	26 CPM
	4-156 keV	29 CPM	22 CPM	20 CPM	19 CPM
	0-1,000 keV	65 CPM	49 CPM	35 CPM	33 CPM

Table 2. Chemiluminescence in cocktails with $Ag^{36}Cl$/ammonia solution

The results mean that the researcher has a choice of two classical cocktails and two safer cocktails. Low energy chemiluminescence will not present a problem in this case, since virtually all of the background counts are in the 0 to 4 keV region and ^{36}Cl is a particularly high energy beta emitter (Emax 709 keV). Therefore, the windows of the LSA can be set to exclude any low energy chemiluminescence.

Recommendation:

1. Use one of the cocktails recommended above.
2. Prepare samples/cocktails in glass vials.
3. If possible, keep samples/cocktails in the dark (solutions of silver salts are decomposed by sunlight and UV light).
4. Set windows to exclude low energy chemiluminescence.
5. Check samples/cocktails after counting to ensure that no decomposition has occurred.

Follow-Up Comment

Two researchers have reported that they are measuring ^{36}Cl successfully using these methods, one is using ULTIMA Gold AB and the other is using ULTIMA Gold LLT.

PerkinElmer, Inc.
940 Winter Street
Waltham, MA 02451 USA
Phone: (800) 762-4000 or
(+1) 203-925-4602
www.perkinelmer.com

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