Determination of Shelf Life for Aldehyde Monitors

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General

Air samplers (monitors) manufactured at Assay Technology are subject to a control system in which an Expiration Date and Lot No. are imprinted at the time of manufacture on each hermetically-sealed foil package containing a single monitor. The Lot No. (from which the Expiration Date can be obtained by look-up) is further imprinted on the body of each monitor.

Expiration Dates have been determined by laboratory studies in which regular stock products (packaged monitors) were held at the storage temperature specified in the Technical Insert, then evaluated by the quality control tests used for freshly-manufactured monitors. (Since monitors are stored in sealed foil pouches, light and humidity storage controls are not necessary.) At Assay Technology, the shelf life is defined as the length of time after manufacture that monitors stored under recommended conditions continue to perform as claimed in the Technical Insert. In some cases, a plot of a performance test versus storage time may be extrapolated to predict a longer shelf life after which monitors would be expected to remain within acceptance limits and perform as claimed. When a shelf life is determined based on extrapolation, studies must be continued to confirm shelf life of stored products.

Background Science

Historically, the Assay Technology 571AT and 581AT monitor has been stored under refrigeration based on earlier studies that suggested increases of background blank on storage.

Formaldehyde is emitted by many industrial and household materials such that concentrations up to 0.02 ppm may be present in ambient outdoor air and even higher levels in some facilities. Accordingly, precautions must be taken to minimize formaldehyde exposure during each stage of monitor manufacture.

In the 1980s, formaldehyde was found in many commercially available packaging materials. Consequently, many shelf life studies on formaldehyde samplers showed an increase in formaldehyde background blank (due to uptake from packaging) during storage. In many cases, the increase in background blank could be reduced by colder storage temperatures, so that refrigerated (and even freezer) storage was recommended for many formaldehyde samplers.

In a never-ending quest to reduce formaldehyde levels in our manufacturing environment and in purchased packaging, Assay Technology acquired packaging that emitted little or no formaldehyde. Stored monitors were then analyzed to measure the amount of active reagent (2,4-dinitrophenylhydrazine) and the amount of formaldehyde background blank in each stored monitor.

Materials & Methods

Monitors were analyzed by extraction followed by high performance liquid chromatography (HPLC) which allows separate analysis of un-reacted reagent (DNPH) as well as analysis of background formaldehyde on the monitor. Monitors from 8 separate manufactured lots analyzed during initial QC, after 1-year of refrigerated storage (2-8°C), followed by 9 months at room temperature (20-30°C) were computed and reported separately as shown.

Results & Conclusions

Table 1 shows the amount of formaldehyde background present on samples taken from each of 8 lots of monitors some analyzed immediately upon manufacture, some after 1 year storage under refrigeration, and some after 1 year of refrigeration followed by 9 months at room temperature storage.

Taking into account random variation, no detectable increase in formaldehyde background can be seen as a result of refrigerated storage, as the average measured increase of 0.001 µg per monitor of formaldehyde is well within experimental variation. When formaldehyde background blanks of monitors analyzed after an additional 9 months' storage at room temperature were compared with those after 1 year refrigerated storage, an average increase of 0.006 µg per monitor was computed. This amount is comparable to the standard deviation among monitors seen during initial QC which (approx. ±0.006 µg). Analysis of data from 6 of 7 lots (removing the worst) led to a computed average increase of only 0.003 µg per monitor.

Conclusions:

(1) No detectable increase in formaldehyde background blank observed during 1 year at 2-8°C.

(2) A non-detectable or possibly detectable increase in formaldehyde blank observed during 9 months' storage at 20-30°C, and all lots did not exceed the claimed limit of 0.050 µg.

We believe these data, including extrapolation to one year's storage, justify recommended storage conditions at Controlled Room Temperature for up to 12 months.

Table 1 - Stability of Background Blank
(Refrigerated & RT Storage)

Lot No.	Initial QC Test	1 Yr Storage (2-8°C)	Amount Increase	9 Mos Storage (20-30°C)	Amount Increase
	(µg HCHO) Ave of 17	(µg HCHO) Ave of 5	(µg HCHO)	(µg HCHO)	
2D17	0.023	0.021	-0.002	0.027	0.006
5B17	0.027	0.031	0.004	0.054	0.023
4I18	0.021	0.027	0.006	0.032	0.005
6C18	0.027	0.023	-0.004	0.032	0.009
8A18	0.032	0.026	-0.006	0.035	0.009
9Q18	0.036	0.034	-0.002	0.032	-0.002
2F19	0.029	0.034	0.005	0.033	-0.001
7A19	0.022	0.031	0.009	0.028	-0.003
		Average (8 lots) =	0.001		0.006
		Average (7 lots excluding 5B17) =	0.001		0.003

Table 2 shows the amount of unreacted DNPH reagent (2,4-dinitrophenylhydrazine) present on monitors taken from each of 8 lots of monitors some analyzed upon manufacture, some after 1 year storage under refrigeration, and some after 1 year of refrigeration followed by 9 months at room temperature storage.

The average amount of un-reacted DNPH reagent found on all monitors stored for 9 months at room temperature (following 1-yr refrigeration) was 101% of the initial QC test result. While individual lots

varied from 92-120% of the amount of DNPH from the initial QC test, we attribute these results to typical variance in DNPH content from monitor-to-monitor. Therefore, our conclusion is that no detectable decrease in DNPH reagent was observed due to storage under refrigeration for 1 year.

The average amount of un-reacted DNPH reagent found on all monitors stored at room temperature for 9 months (following 1-yr refrigeration) was found to be, 93% of the average of initial QC tests with the range of 85-106% for individual lots. Despite monitor-to-monitor variation, we believe this may suggest a detectable decrease in DNPH reagent on the monitor.

The amount of DNPH reagent on each monitor is designed to be well in excess of what is required to react with formaldehyde collected, so that claimed capacity of 12.5 ppm formaldehyde for an 8-hr sample is maintained so long as DNPH is within $\pm$ 25% of the average 900 µg ($\pm$ 225 µg) observed in these tests.

Our conclusion from these data are as follows.

(1) No detectable decrease of DNPH reagent was observed during 1 year storage at 2-8°C.

(2) A possibly detectable decrease of 7% in DNPH reagent observed during 9 months' storage at 20-30°C, but still within the amount required to provide the claimed capacity to collect formaldehyde vapor.

(3) Extrapolation predicts the monitor would retain sufficient DNPH reagent after 12 months storage.

We believe these data, including the data on formaldehyde background blank, justify recommended storage conditions at Controlled Room Temperature for up to 12 months.

Table 2 - Stability of Active Reagent DNPH
(Refrigerated & RT Storage)

Lot No.	Initial QC Test	1 Yr Storage (2-8°C)	% of Initial QC	9 Mos Storage (20-30°C)	% of Initial QC
	(units DNPH)	(units DNPH)	(%)	(units DNPH)	(%)
	Ave of 17	Ave of 5			
2D17	861	949	110%	887	103%
5B17	821	988	120%	867	106%
4I18	984	920	93%	832	85%
6C18	934	942	101%	858	92%
8A18	951	1004	106%	853	90%
9Q18	973	870	89%	827	85%
2F19	859	836	97%	812	95%
7A19	924	846	92%	830	90%
		Average (8 lots) =	101%		93%