Validation of Ethanol

Using SKC Cat. No. 575-002 Diffusive Samplers

Research Report

Validation of Ethanol Using SKC Cat. No. 575-002 Diffusive Samplers

Abstract

A sampling method using the SKC Cat. No. 575-002 diffusive sampler has been partially validated for sampling ethanol in workplace air. A previously validated desorption efficiency (DE) of 99% (2013 SKC Inc. Catalog) was used in this validation. The sampling rate was determined for Cat. No. 575-002 samplers exposed to ethanol levels of 114 ppm and 880 ppm at 60% relative humidity (RH) and 25 C. The mean sampling rate for

32 tests was 20.3 ml/min with a 4.02% RSD. Cat. No. 575-002 samplers can be stored at ambient or freezer temperatures (-22 C) for up to three weeks with a 10% loss in recovery or less. All diffusive samplers were desorbed in 2 ml of 10% 2-butanol in carbon disulfide and analyzed by gas chromatography with flame ionization detection (FID).

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Introduction

Ethanol, also known as ethyl alcohol, absolute alcohol, grain alcohol, anhydrous alcohol, dehydrated alcohol, and ethyl hydrate, is a clear flammable liquid with a pleasant odor and burning taste. Ethanol is manufactured by the fermentation of starch, sugar, and other carbohydrates and also from ethylene, acetylene, sulfite waste liquors, and by hydrolysis of ethyl sulfate and oxidation of methane. Ethanol is mostly used in alcoholic beverages, in the manufacture of denatured alcohol, pharmaceuticals such as rubbing compounds, lotions, and tonics, in perfumery, organic synthesis and as an octane booster in gasoline. (1) Ethanol was used as lamp fuel in the United States as early as 1840, but a tax levied on industrial alcohol during the Civil War made this use uneconomical. Original Ford Model T automobiles ran on ethanol until 1908. (2) When Prohibition started in 1920, ethanol fuel sellers were accused of working with moonshiners (3) and ethanol fuel fell into disuse until late in the 20th century. Overexposure to ethanol may cause nausea, vomiting, flushing, mental excitement or depression, drowsiness, impaired perception, incoordination, stupor, coma, or death. Ethanol may have reproductive and fetal effects. (1)

The purpose of this study is to validate the Cat. No. 575-002 diffusive sampler for monitoring ethanol at 100 ppm and 1000 ppm.

Experimental

Ethanol (Sigma Aldrich, St. Louis, MO, U.S.A.) was used to prepare concentrations in the test rig. A dynamic atmosphere was generated using a syringe pump and filtered air streams to generate the concentration. The system is shown in Figure 1. The atmosphere was fed into an exposure chamber and the diffusive samplers were exposed on a rotating bracket inside the chamber to simulate wind velocity. The sampling rate was conducted at 880 ppm for two hours and at 114 ppm for periods from 15 minutes to 8 hours at 60% RH and 25 C. The concentration within the atmospheric chamber was verified with Cat. No. 226-01 sorbent tubes (SKC Inc., Eighty Four, PA, U.S.A.). The Cat. No. 575-002 diffusive sampler (SKC Inc., Eighty Four, PA, U.S.A.) was used for the study. After exposure, samplers were sealed until analysis.

The storage study consisted of injecting 21 Cat. No. 575-002 passive samplers (SKC Inc., Eighty Four, PA, U.S.A.) with known amounts of ethanol. The samplers were capped and allowed to equilibrate overnight. Three samplers were analyzed the next day, while nine samplers were stored at ambient temperatures and the remaining nine samplers were stored in a freezer (-22 C). Three samplers were analyzed each week for three weeks from both temperatures to determine the analytical recovery.

All diffusive samplers were desorbed in 2 ml of 10% 2-butanol in carbon disulfide and shaken on a flatbed shaker for 15 minutes. The extracts were then analyzed by flame ionization detection gas chromatography. A chromatogram is shown in Figure 2.

SKC Inc. constantly reviews this data and conducts experiments to provide the most precise sampling rate. The rate published in these validation reports is the correct rate.

Results and Discussion

The desorption efficiency used in this validation was 99% (2013 SKC Inc. Catalog) when used with the Cat. No. 575-002 passive diffusive sampler. The sampling rate data is shown in Table 1. The results of the 32 samplers show that ethanol can be sampled with Cat. No. 575-005 diffusive samplers at an average sampling rate of 20.3 ml/min (4.02% RSD). The data indicates that the sampler can collect a 15-minute to 8-hour sample at 114 ppm of ethanol and a 2-hour sample at 880 ppm. The three week storage study, shown in Table 2 and

Table 3, suggest that samplers are able to be stored at either ambient temperatures or in a freezer for three weeks with a 10% or less loss in recovery.

Conclusion

Cat. No. 575-002 diffusive samplers have been partially validated for sampling ethanol with a DE of 99% and sampled at a rate of 20.3 ml/min (4.02% RSD). The samplers showed good stability when stored for three weeks at both ambient and freezer temperatures with a 10% or less loss in recovery. Cat. No. 575-002 diffusive samplers can be used for measuring exposes of ethanol from 15 minutes to 8 hours at 114 ppm and 2 hours at 880 ppm.

Reference

1. *Merck Index, 12th Edition, p. 643*
2. *Siegel, Robert, "Ethanol, Once Bypassed, Now Surging Ahead," (2007-02-15), NPR, http://www.npr.org/templates/story/story.php?storyId=7426827. Retrieved 2012-05-24*
3. *DiPardo, Joseph, "Outlook for Biomass Ethanol Production and Demand" (PDF), United States Department of Energy, http://tonto.eia.doe.gov/FTPROOT/features/biomass.pdf. Retrieved 2012-05-24*

Table 1. Sampling Rate
Ethanol
114 ppm (0.25 to 8.00 hrs, 880 ppm (2.00 hrs), 60% RH, 25 C

Time (hr)	Sampling Rate (ml/min)
0.25	19.5
	20.0
	19.5
	20.6
0.50	21.2
	21.5
	21.1
	20.8
1.00	20.2
	20.1
	21.0
	20.5
2.00	19.9
	20.3
	21.9
	20.2
	20.1
	19.6
	20.4
	19.2
4.00	21.1
	19.4
	21.2
	20.9
6.00	19.5
	20.6
	18.8
	20.6
8.00	19.3
	18.3
	20.2
	20.3
Mean	20.3 ml/min
Std. Dev.	0.813
RSD	4.02%

Table 2. Storage Study
Ethanol, Ambient Temperatures

Week	Recovery (%)
1	98.7
2	96.7
3	90.3

Table 3. Storage Study
Ethanol, Freezer Temperatures

Week	Recovery (%)
1	101
2	101
3	96.9

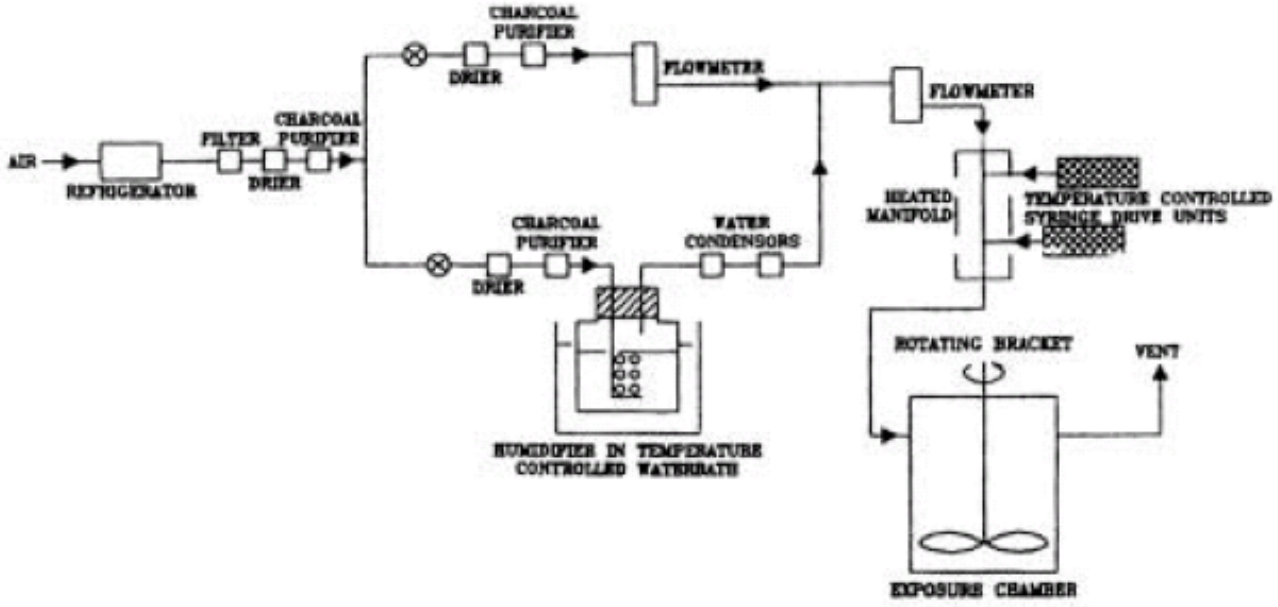


Figure 1. Test System

Appendix A

Atmosphere Generation Apparatus

The instrument is designed to expose a known concentration of a chemical hazard to a passive sampler under controlled conditions of: 1. Concentration, 2. Temperatures, 3. Humidity, 4. Wind Velocity Effect, and 5. Time.

Description

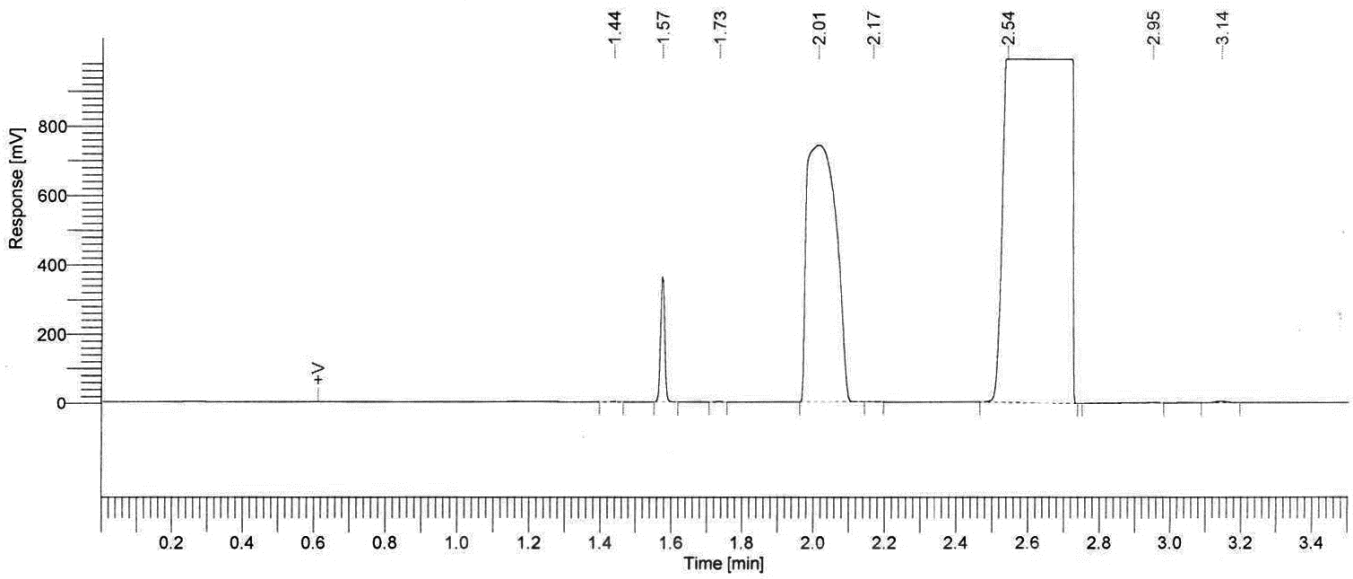
The instrument consists of:

1. An exposure chamber in which the wind velocity effects are controlled by internal rotating holders.
2. An air supply and purification train such that dry air is blended with saturated air under desired temperature conditions so as to provide air known at a known flow and selectable humidity.
3. An injection system composed of a precision motor driven syringe in which the chemical hazard can be injected into the flow system and in which the temperature of the injector is closely controlled.
4. An electrical control system that controls the entire instrument operation.
5. The chamber concentration can be verified by either solid sorbent sampling tubes actively sampled or by gas analysis of the gas phase. The particular verification method used will depend on the analyte of interest.

Means are also included to check the relative humidity.

Figure 2

Sample Chromatogram
Ethanol



Ethanol

Column: RTX-5, 30 m x 0.32 mm ID, 1 micron film

Temperatures:

Column: 50 C isothermal

Injector: 250 C

Detector: FID at 250 C

Retention Times

Ethanol	1.57 minutes
Carbon disulfide	2.01 minutes
2-Butanol	2.54 minutes