(conoco)

Technical Service Report

Conoco Inc.
Research and Development Department
Analytical Research Section
Ponca City, Oklahoma

Report No 435-81-839-9

To E. L. Sones

From C. B. Herrin

Date April 22, 1981

Subject GC/MS EXAMINATION OF TWO LAKE CHARLES EFFLUENT WATERS FOR ACROLEIN AND ACRYLONITRILE

Conclusion: Neither of the title pollutants was found in either sample.

Introduction

This is a continuation of the work described in TSR 429-81-804-8. The samples examined were 4236-93-12 from the chemical plant and 4236-93-2 from the VCM plant. Both were stored in the cold room until shortly before they were run. Both samples were run on the fifteenth of April.

Experimental

Preparation of the Analytical Column:

An empty glass column ~6' x 2 mm i.d. was thoroughly cleaned and dried. It was silanized by filling with "Sylon" and allowing it to stand at room temperature for five minutes. The "Sylon" solution was removed by suction, and the tubing was washed with toluene and anhydrous methanol. Air was pulled through until the methanol had evaporated, and then the empty column was installed in a chromatograph and heated to 150°C with He flow. The packing, Chromosorb 101 80/100 mesh, was preconditioned overnight in a large diameter metal column at 180°C with He flow. The packing was dumped from the large column and resieved. The glass column was packed, and the ends were plugged with silanized glass wool. The column was installed in the Sigma III (GC/MS chromatograph) oven with the column exit disconnected and conditioned overnight at 150°C.

Instrumental Conditions

Sample - on-column injection of 5 µℓ
Injector - 130°C
Column - 130°C
Carrier Gas - He @ 20 cc/min
Scan - 20 AMU to 100 AMU in 4 sec.

Procedure

Standards of acrolein and acrylonitrile were injected to determine retention times and obtain good quality spectra. Detection limits were estimated by injecting aqueous standard solutions and obtaining mass chromatograms at 53 and 56 AMU. The samples were run, and the reconstructed ion and selected ion chromatograms were examined.

Discussion of Results

The detection limits were about the same as those reported in TSR 429-81-804-8, and these limits are determined by the level of interferences rather than instrumental limitations. When water elutes from a column, it apparently "steam strips" organic impurities along with it. This is a commonly observed phenomenon in gas chromatography. In this case the elution of water generated a very broad peak which encompassed the elution range of acrolein and acrylonitrile. The spectra of the organic peaks throughout the broad peak were similar to background spectra taken before and after the peak. The generation of this peak seemed to result from a general increase of background intensities. These spectra contained peaks at every mass number. Therefore, the mass chromatograms, regardless of the ion selected, had a high noise background and the same general appearance as the reconstructed ion chromatogram.

Mass chromatograms at 53 and 56 AMU, the base peaks for acrylonitrile and acrolein, did not reveal any peaks above the noise background. The amounts present, if any, are certainly less than 1 ppm for either pollutant. In this work there was no evidence of a peak with the same retention time as acrolein (see TSR 829-81-804-8). The peak observed with the FID was probably an artifact peculiar to the chromatographic system used.

Reference: TSR 829-81-804-8

C. B. Herrin

C. B. Herrin
Research Group Supervisor
Gas Chromatography Group
Analytical Research Section

BW
Copy to Library