United States
Environmental Protection
Agency

Atmospheric Research and
Exposure Assessment Laboratory
Research Triangle Park, NC 27711

Research and Development

EPA/600/SR-93/229 February 1994

EPA

Project Summary

Closed Cycle Cooler for VOC Preconcentration

Deborah L. Smith

The objective of this study was to evaluate a reduced temperature preconcentrator to replace or reduce liquid cryogen use in field applications. The cooler was to be evaluated as a refocusing unit downstream of a solid sorbent trap. A closed cycle cooler from Cryoworks (formerly Carlisle Cryotronics) in Carlisle, MA, was selected for the evaluation. Basic properties of the cooler were evaluated, including cooldown time and heating time. Modifications were made to improve the performance of the unit during the testing period. Details of the study are presented.

This Project Summary was developed by EPA's Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, NC, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

The use of automated gas chromatographs at air quality network monitoring stations to obtain concentrations of volatile organic compounds (VOCs) has been a long-term goal of the methods development group at the U.S. Environmental Protection Agency. Developmental efforts by EPA and other research laboratories have resulted in several prototype units capable of measuring VOCs in ambient air. These prototype units generally utilize a solid adsorbent trap to adsorb VOCs from an air stream followed by thermal desorption onto a liquid cryogen cooled refocusing trap. The refocusing trap is in turn thermally desorbed and the sample is sent to a capillary column for resolution of the components. In some instances the capillary column is also subambiently cooled to better facilitate compound resolution. These automated gas chromatographs are capable of measuring a wide range of VOCs in ambient air that include C_2 through C_{10} hydrocarbons, as well as select halogenated and oxygenated organics. To effectively retain and resolve the C_2 hydrocarbon species on a single column, a considerable amount of liquid cryogen is required. Most systems cool the adsorbent and the refocusing traps as well as the analytical column by controlled release of liquid cryogen. The use of cryogen becomes a significant disadvantage when deploying the automated systems in field monitoring studies. That is, the frequent delivery of liquid cryogen required, and the uneven quality of the product typically delivered, often result in manpower commitments and, to a certain extent, costs that detract from the usefulness of the systems.

In an attempt to circumvent the use of liquid cryogen, an alternative approach that involved the use of a closed cycle cooling device was investigated. Cryoworks (formerly Carlisle Cryotronics) designed and produced a Gifford-McMahon style of closed cycle cooler that incorporates mechanically operated valves to control gas flow. Cryoworks had recently developed a magnetic stroke technology in their cooling engines for controlling the movement of a gas driven, piston actuated displacer (patent pending). Other commercial units normally employ mechanically driven engines. Increased operating efficiency as well as enhanced long-term reliability were key attributes of the Cryoworks technology.

Printed on Recycled Paper

Objective

The objective of this study was to evaluate the Cryoworks* closed cycle cooler as a refocusing unit downstream of a solid adsorbent trap. Initial tests focused on determining the system's capabilities in terms of its cooling and heating efficiency. Target time and temperature conditions were determined based upon previous work with automated refocusing units that require liquid cryogen to retain C_2 hydrocarbons.

Evaluation

The Cryoworks closed cycle cooler was evaluated as a refocusing unit downstream of a solid adsorbent trap. Tests were performed to determine the system's capabilities in terms of cooling and heating efficiency using target time and temperature conditions. For optimum performance the Cryoworks cooler was required to reach and maintain an initial temperature of at least -180°C. The system was also required to heat to 150°C within 30 seconds. Finally the system was required to return to -180°C within 20 min. Initial evaluation included testing the system in its original configuration as received from Cryoworks. Further tests were performed after modifications were made to the cold station.

During initial evaluation of the Cryoworks closed cycle cooler the system was able to reach -178°C after 2 hours but was not able to maintain the temperature for any extended period of time, indicating erratic and unreliable behavior. Modifications were made to the Cryoworks unit at Battelle to improve its performance. These changes resulted in allowing the cooler to be cycled reliably to temperatures as low as -197°C as well as the desired thermal cycling can be repeated in as little as 14 min, well within the requirements of operation of a gas chromatograph for automated sampling and analysis. Furthermore, the rise time for heating was approximately 0.4 min, which also meets the requirements for the closed cycle cooler. Unfortunately, evidence of a small hotspot area was seen just outside of the cold zone. The thermocouple was monitoring the temperature near this area and was not giving a true indication of the temperature within the cold zone. Repeated attempts to improve this situation were unsuccessful.

Conclusions and Recommendations

In its original configuration from Cryoworks, the closed cycle cooler was unable to reach the temperature setpoint of -180°C during initial cooldown. After modifications were made by Battelle to the cold station, the system was able to reach -180°C in less than 90 min. Once this temperature was achieved, thermal cycling tests were performed to simulate the use of the system in repetitive operation as an auto GC. During these tests, the unit was able to reach approximately 150°C in 0.4 min and return to -180°C in 14 min. This is well within the target values set for the system. We believe that during the initial test cycles the thermocouple was actually monitoring the temperature near a hotspot and thus was giving an erroneous indication of the temperature within the cold head area. Efforts to eliminate the hotspot and better control the temperature were not successful.

The general impressions of the Cryoworks closed cycle cooler are mostly favorable. The system definitely has the capability of cooling a section of fused silica to -180°C and below, and we believe the system has additional capacity to provide cooled air to reduce the temperature of the primary sample collection trap and the gas chromatographic column of an automated system. The cooler can be mounted easily onto a Hewlett-Packard Model 5890 gas chromatograph directly above the heated injection ports. This configuration allows for a smooth transition from the fused silica refocusing trap to the analytical column.

We believe that the Cryoworks closed cycle cooler would be a very effective reduced temperature preconcentrator to be used as a refocusing unit downstream of a solid adsorbent trap. We have demonstrated that the system meets the target time and temperature requirements needed for cooling a section of fused silica for collection of light hydrocarbons. We recommend that further improvement to the system is needed to optimize its performance in the heating mode.

This report was submitted in fulfillment of Contract No. 68-DO-0007 by Battelle's Columbus Operations under the sponsorship of the U.S. Environmental Protection Agency. This report covers WA-7 and WA-16 over a period from November 1990 to August 1992, and work was completed as of August 15, 1992.

*Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

☆U.S. GOVERNMENT PRINTING OFFICE: 1994 - 550-067/80192

Deborah L. Smith is with Battelle, Columbus, OH 43201.
***William A. McClenny** is the EPA Project Officer (see below).*
The complete report, entitled "Closed Cycle Cooler for VOC Preconcentration," (Order No. PB94-130 390/AS; Cost: $17.50, subject to change) will be available only from:

National Technical Information Service
5285 Port Royal Road
Springfield, VA 22161
Telephone: 703-487-4650

The EPA Project Officer can be contacted at:

Atmospheric Research and Exposure Assessment Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

United States
Environmental Protection Agency
Center for Environmental Research Information
Cincinnati, OH 45268

BULK RATE
POSTAGE & FEES PAID
EPA
PERMIT No. G-35

Official Business
Penalty for Private Use $300

EPA/600/SR-93/229