Oxygen Carrier for Gas Chromatographic Analysis of Inert Gases in Propellants

The gas chromatographic determination of small quantities of inert gases dissolved in reactive propellants is not easily performed with carrier gases such as helium, nitrogen, or hydrogen. For example, the determination of helium in diborane with thermal conductivity detectors is complicated by traces of water in column packing materials, because hydrogen is formed by the hydrolysis of diborane, and the separation of hydrogen from helium is not readily accomplished on any column of convenient length. Moreover, hydrogen is not a satisfactory carrier gas because its thermal conductivity is nearly the same as helium. Oxidation of diborane by a hot cupric oxide bed placed in front of the gas chromatograph column proved to be only partially effective; however, when oxygen was used as the carrier, complete oxidation of the diborane was obtained and satisfactory determinations of 0.09 to 1.5 mol-percent of helium were obtained.

In another instance, the determination of nitrogen in oxygen difluoride by the usual gas chromatographic technique with helium as a carrier gas was unsuccessful because the oxygen which is produced by reaction of the propellant with column materials is not readily separated from nitrogen by the chromatographic columns ordinarily employed. However, it was found that oxygen could be used effectively as a carrier with glass-bead thermistor detectors, but the sensitivity of the determination was somewhat reduced.

Operating conditions used for these specific analyses are given in the table.

<table>
<thead>
<tr>
<th>Conditions</th>
<th>He in B₂H₆</th>
<th>N₂ in OF₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carrier gas</td>
<td>Oxygen</td>
<td>Oxygen</td>
</tr>
<tr>
<td>Flow rate</td>
<td>40 ml/min</td>
<td>40 ml/min</td>
</tr>
<tr>
<td>Column 1)</td>
<td>50.8-cm long, 60-80 mesh silica gel pre-conditioned with moist air; 2) 25.4-cm long, CuO wire at 704°C; 3) 15.2-cm long, Linde 13X molecular sieve, 40-60 mesh.</td>
<td>50.8-cm long, Linde 13X molecular sieve + 20.3 cm of soda lime</td>
</tr>
<tr>
<td>Column temperature</td>
<td>Room temperature; 2) 704°C; 3) 38°C</td>
<td>38°C</td>
</tr>
<tr>
<td>Sample size</td>
<td>0.5 to 1.0 ml</td>
<td>0.5 to 2.0 ml</td>
</tr>
</tbody>
</table>

Notes:
1. The following documentation may be obtained from:
   National Technical Information Service
   Springfield, Virginia 22151
   Single document price $6.00
   (or microfiche $0.95)
Reference:
NASA CR-97860 (N69-10713), Liquid Propellant Gas Absorption Study.

Patent status:
No patent action is contemplated by NASA.
Source: William A. Cannon of McDonnell Douglas Corporation under contract to Ames Research Center (ARC-10574)

2. No additional documentation is available. Specific questions, however, may be directed to:
Technology Utilization Officer
Ames Research Center
Moffett Field, California 94035
Reference: B72-10249