During a test, all measurements are recorded by use of a portable data-acquisition system and a computer. The total heat-leak rate is measured and calculated as the boil-off flow rate multiplied by the latent heat of vaporization. The parasitic heat leak (to the side of the specimen and to the top and side of the cold-mass tank) is reduced to a small fraction of the total heat leak by use of a combination of multilayer-insulation (MLI) shield rings, reflective film, a fiberglass/epoxy centering ring, and a bulk fill of aerogel beads. This combination eliminates the need for a cryogenic guard chamber used in a typical prior apparatus to reduce the parasitic heat leak.

This work was done by James E. Fesmire of Kennedy Space Center and Stanislaw D. Augustynowicz of Dynacs, Inc. Further information is contained in a TSP (see page 1).

KSC-12390

Quadrupole Ion Mass Spectrometer for Masses of 2 to 50 Da

H₂, He, O₂, and Ar can be quantitated at low concentrations in N₂.

John F. Kennedy Space Center, Florida

A customized quadrupole ion-trap mass spectrometer (QITMS) has been built to satisfy a need for a compact, rugged instrument for measuring small concentrations of hydrogen, helium, oxygen, and argon in a nitrogen atmosphere. This QITMS can also be used to perform quantitative analyses of other gases within its molecular-mass range, which is 2 to 50 daltons (Da). (More precisely, it can be used to perform quantitative analysis of gases that, when ionized, are characterized by m/Z ratios between 2 and 50, where m is the mass of an ion in daltons and Z is the number of fundamental electric charges on the ion.)

The QITMS was assembled mostly from commercial components. It includes a vacuum manifold, ion trap, and filament from a commercial ion-trap mass analyzer that produces ions within the ion trap by electron impact. Low-molecular-weight ions are effectively trapped when internal electron impact is used, despite the absence of a collision (buffer) gas. This internal-electron-impact assembly is compact, rugged, and suited for use in a miniaturized instrument. The assembly was modified to (1) increase the opening of the trap to the vacuum manifold in order to increase the rates of transport of analyte gases, (2) accommodate an ion high-vacuum gauge that measures the pressure in the open ion trap, and (3) replace an original gas-chromatograph-type transfer tube with a stainless-steel tube for introducing gas samples into the trap. A high vacuum is produced in the trap by means of a turbo-drag pump backed by either a diaphragm pump or a rotary-vane pump.

The electronic control and monitoring unit of the aforementioned commercial ion-trap mass analyzer was replaced by a more capable control and monitoring unit from another commercial ion-trap mass analyzer, with minor changes in the electron-filament portion of the replacement unit to regulate emission of electrons from the filament. With this modification, the electron-emission assembly produces a 200-µA current of electrons at a kinetic energy of 90 eV. An electron multiplier from the second-mentioned commercial ion-trap mass analyzer was in-

This Mass Spectrum was obtained in an initial test of the QITMS, using a mixture of H₂, He, O₂, and Ar, each at a concentration of 1.25 percent in N₂.
installed in the vacuum manifold to match an electrometer circuit in the control and monitoring unit.

The instrument operates under control by a computer that runs custom software. Included in the software is a module for performing real-time monitoring of selected gases at a chosen update rate (e.g., once per second).

Like commercial instruments, this QITMS utilizes a mass-selective instability for mass analysis. Both commercial ion-trap analyzers from which the parts were taken to build the present unit have low-mass cutoffs of 4 Da. To extend the lower mass limit to 2 Da with the least amount of modification and fabrication, it was decided to increase the upper limit of frequency of the signal applied to coils to generate the trapping radio-frequency field. This decision was implemented through modifications of the signal-generating circuits and construction of replacement coils to provide multiple resonance frequencies from 1 to 4 MHz. Increasing the upper limit of frequency reduced the upper limit of the mass range below that of the unmodified commercial instruments, but this was acceptable because the upper mass limit of 50 Da required for this instrument remained within range.

The QITMS was initially tested at a frequency of 2.8 MHz with a sample gas mixture comprising 1.25 percent, each, of hydrogen, helium, oxygen, and argon in nitrogen. The results of this test showed readily identifiable ion signals at m/z values of 2, 4, 32, and 40, with an upper m/z limit only a few tenths above 40 (see figure). In a subsequent test, it was found that the desired m/z range of 2 to 50 could be attained in operation at a frequency of 2.5 MHz. In other tests, it was found that the relative accuracy and precision in quantitating the four gases of interest were characterized by an error of no more than 10 percent of reading and a deviation of no more than 5 percent of reading, respectively. In still other tests, it was found that the lower limits of detectable concentrations were 25 parts per million (ppm) for hydrogen, 100 ppm for helium, slightly higher than 25 ppm for oxygen, and slightly higher than 10 ppm for argon.

This work was done by William Helms of Kennedy Space Center; Timothy P. Griffin of Dynacs, Inc.; and Andrew Ottens and Willard Harrison of the University of Florida. Further information is contained in a TSP (see page 1). KSC-12428