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Secondary Ion Mass Spectrometry: a Tool for Identification of Matrix-isolated Species

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Summary An argon matrix-isolated propane sample (1:150, 10 K) is used to demonstrate the applicability of secondary ion mass spectral analysis to the characterization of matrix-isolated species.

LOW-TEMPERATURE matrix isolation has become an important technique for the preparation and study of reactive species.¹ We now report that secondary ion mass spectrometry (SIMS), commonly used by surface analysts,² can be used to obtain a mass spectral fragmentation pattern for a molecule isolated in an argon matrix at 10 K.

In SIMS, ions for mass spectral analysis are produced by a beam of energetic primary ions which sputters and ionizes particles off a solid surface. An electron flood gun is used to neutralize surface charges if the solid is an insulator. We find that the argon matrix suffers no macroscopic damage, but its surface appears to be slowly etched away by the bombarding primary beam, permitting depth profiling.

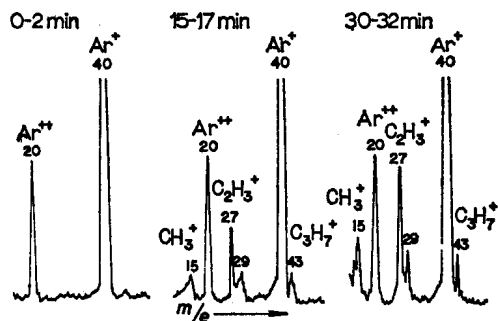


FIGURE 1. SIMS of propane in argon matrix (1:150), 10 K, ${}^3\text{He}^+$, 1 keV.

Figure 1 shows the results of successive single two-minute SIMS scans of solid propane-doped argon as obtained on a modified 3M Company Model 525 spectrometer (ca. 1×10^{-8}

Torr background pressure; controls were run to ensure that the signal does not originate in the vapour phase). After the argon-rich surface layer is sputtered away, no further change is observed in the ratio of the propane and argon ion intensities, illustrating the depth-profiling inherent to the method. The SIMS fragmentation pattern is the same for more concentrated and even for neat propane (Figure 2), and contains fragment ions at the same m/e

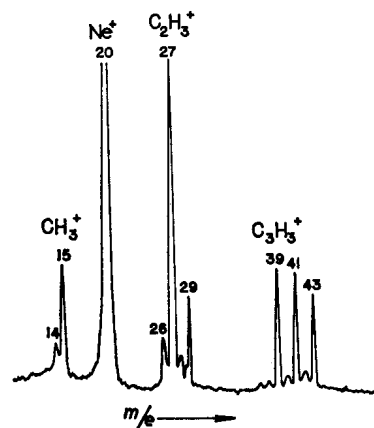


FIGURE 2. SIMS of solid propane (a single ten-minute scan), 35 K, ${}^{20}\text{Ne}^+$, 1 keV.

values as the ordinary electron-impact mass spectrum of propane, but with a different intensity distribution. The most striking difference is the absence of the molecular ion peak at m/e 44. In general, we find the low-temperature SIMS results to be independent of the nature and kinetic energy of the primary ions and of temperature within the limits permitted by the nature of the sample. In neat solid hydrocarbons, ion-molecule reactions apparently occur under SIMS conditions, as evidenced by the occurrence

of fragments heavier than the molecular ion, but such complications have not been observed in the argon matrix isolation experiments.

In summary, doped argon matrices easily withstand SIMS conditions and yield characteristic fragmentation patterns which promise to be useful as a structural identification tool for the dopant.

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¹ 'Cryochemistry,' eds. M. Moskovits and G. A. Ozin, Wiley, New York, 1976.

² A. Benninghoven, *CRC Crit. Rev. Solid State Sci.*, 1976, 291; *Surface Sci.*, 1973, **35**, 427; H. W. Werner, *ibid.*, 1975, **47**, 301; *Vacuum*, 1974, **24**, 493.