

## Chemometrics: Theory and Application

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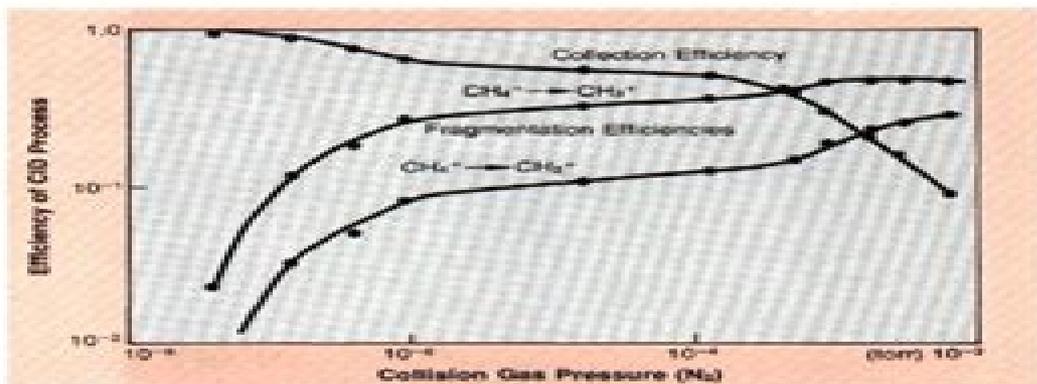


Figure 6. Effect of collision gas (N<sub>2</sub>) pressure on CID efficiency for methane CH<sub>4</sub><sup>+</sup> at ion initial energy of 10eV

chemical and electrical noise levels, and the lowest measurable signal level. The electrical noise in the system is typically  $3 \times 10^{-13}$  A. The two stages of mass separation often make it possible to reduce chemical noise (ions detected from other than the desired reaction) to well below the  $10^{-13}$  A level. In such a case, at the extreme sensitivity limit, the peak height is quantized by the integer number of ions which reach the detector during the scanning time of the peak. The detection of 4 ions would give an S/N of 2 and, for methane, would require 6 fg of sample. The detection limit for methane has been determined experimentally to be 16 femtograms by measuring a sample of 10 ppm CH<sub>4</sub> in N<sub>2</sub> at a sample flux of 30 femtograms per sec of CH<sub>4</sub>. The large excess of N<sub>2</sub> is required to increase the ion source pressure so that it can be accurately measured and the sample flux calculated. The selectivity of monitoring the CH<sub>4</sub><sup>+</sup> → CH<sub>3</sub><sup>+</sup> reaction has effected a two order of magnitude improvement in detection limit compared to that obtained by simply monitoring the CH<sub>4</sub><sup>+</sup>, CH<sub>3</sub><sup>+</sup>, or CH<sub>2</sub><sup>+</sup> ions from electron impact. This improvement is due to the elimination of the chemical noise introduced by background <sup>14</sup>N<sup>+</sup>, <sup>15</sup>N<sup>+</sup>, and <sup>16</sup>O<sup>+</sup> peaks.

The selected ion fragmentation capability of the instrument produces only a small loss in transmission and sensitivity, and produces a significant gain in selectivity. Indeed, in the normal case where the system is limited by chemical rather than electrical noise (3), a substantial improvement

in detection limit can be achieved as was observed for methane. The detection limit of approximately 10<sup>-13</sup> mole is made possible by the high efficiency of every component of the system.

The detection limit for a higher mass organic compound, nitrobenzene, has also been determined experimentally. The transition NO<sub>2</sub>C<sub>6</sub>H<sub>5</sub><sup>+</sup> → C<sub>6</sub>H<sub>5</sub><sup>+</sup> was monitored at unit mass resolution in both mass filters, with the parent ion produced by electron impact on a sample of 10 ppm nitrobenzene in N<sub>2</sub>. A detection limit of 120 femtograms (S/N = 2) was obtained with a sample flux of 200 femtograms/sec. In comparison, for the MIKES technique, Cooks has estimated a detection limit of 10 pg for the 140<sup>+</sup> → 123<sup>+</sup> transition of protonated nitrophenol ions produced by CI (22). Although consumption of only femtograms is required for recording the single reaction, actual sample size is significantly larger than this and is dependent on the sample inlet used. Total consumption of a sample introduced by direct probe should make it possible to detect quantities of individual components at close to these detection limits.

### Conclusions

The CID process performed in the strong focusing field of an RF-only quadrupole is efficient and effective in producing characteristic spectra of selected ions. The kinetic energy independence of the mass resolution is ideal for collision product analysis. The lack of interdependence in the control of the two mass filters makes

# Chemometrics Theory And Application

**AW Chickering**



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