ALKANE LADDER CHARACTERISATION FOR RETENTION TIME CALIBRATION USING GC-MS LOW ENERGY EI

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INTRODUCTION

In Gas Chromatography, retention indexes (RI) are used to convert retention times into system-independent constants. Retention index for a compound is calculated normalising its retention time to the retention time of adjacently eluting n-alkanes. While retention times vary with system and method conditions (e.g. carrier gas flow, temperature ramp), the derived retention indices are not affected, allowing comparison of data acquired by different analytical laboratories under varying conditions. EI spectra libraries store retention indices information for several compounds. Deconvolution and library search platforms can use that information as an additional confirmation point when assigning ID to unknowns. Agilent MassHunter Unknowns Analysis software offers a retention time (RT) calibration option which calculates the components retention indices on the basis of the alkane ladder run with the same instrumental conditions. The calculated retention indices are then compared with the library to aid correct matching. To perform the RT calibration, an alkane ladder needs to be acquired by the same method used for the samples. Identification of the different alkanes can be challenging due to the high similarity of the EI spectra and the low abundance of the molecular ion. This often leads to misasignment during library search as well. Using low energy EI to acquire the alkane ladder can easily help with the identification of the molecular ion hence assignment of the right ID to the selected alkane.

INSTRUMENTATION

Autosampler: Dual head MPS Robotic, USM tool equipped with 10 µL syringe
GC-MS: Agilent GC 7890B coupled to Agilent 7250 Q-TOF MS, Low energy EI source

Figure 1: GERSTEL Dual Head Robotic MPS on top of Agilent GC 7890B coupled to 7250 Q-TOF MS

RESULTS

Figure 2 and Figure 3 show the TIC peaks and relative MS spectra, respectively, using Standard EI (black) and Low Energy EI (red) for C19 of the alkane ladder (i.e. Nonadecane).

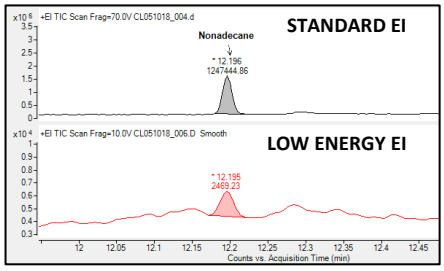

Figure 2: Alkane ladder TIC detail for Nonadecane in Standard EI (top) and Low energy EI (bottom)

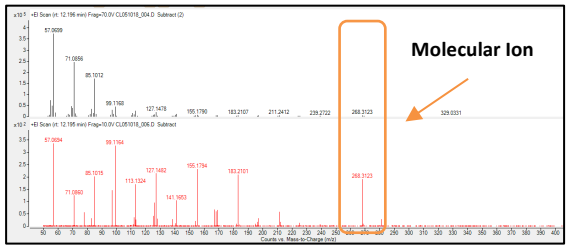

Figure 3: Standard EI (top) and Low Energy EI (bottom) for Nonadecane

NIST search of the standard EI spectrum provided Heneicosane as first match, leaving Nonadecane only as 6th candidate as shown in Figure 4.

#	Lib.	Match	R.Match	Prob. (%)	RI	Name
1	R	853	901	7.23	2100	Heneicosane
2	R	852	895	7.23	2100	Heneicosane
3	M	849	897	6.10	2000	Eicosane
4	R	847	903	5.63	1800	Octadecane
5	R	847	894	5.63	2700	Heptacosane
6	R	846	926	5.41	1900	Nonadecane
7	R	844	884	4.99	2400	Tetracosane

Figure 4 : NIST search matches for the standard E spectrum of Nonadecane in the alkane ladder

The low energy EI spectrum tilts significantly towards the molecular ion providing a crucial piece of information for the identy confirmation of Nonadecane (Molecular ion ID and Accurate Mass, in this case < 1ppm).

CONCLUSIONS

The use of low energy EI spectra can help considerably with the identification of the alkane ladder to build an RT calibration file for the calculation of component RI.

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