

Enhanced Simultaneous Total Ion and Selective Multi-Ion GC/MS Using a Combination of New Software and Low-Bleed Columns

Martin Okiro and Robert Wohleb

VICI Gig Harbor Group Inc, P.O. Box 2657, Gig Harbor, WA 98335

Key Words: GC/MS; Enhanced MSD (E-MSD); simultaneous single ion monitoring

Summary

A new mass spectrophotometer (MS) data acquisition system comprised of hardware, firmware and software has been developed to tune, calibrate and acquire data on single or quadrupole mass spectrometers. The data acquisition routine is so flexible and powerful that several data collection methods can be utilized during the course of a single run. For instance, total ion chromatography and single-ion monitoring can be accomplished simultaneously. Consequently, with a single injection, a total ion chromatogram (TIC) can be generated as well as additional chromatograms each targeting up to 32 individual ions. A combination of this enhanced MSD and exceptionally low-bleed columns results in a powerful new analytical tool allowing detection limit improvement by a factor of ten on EPA method 8270 semivolatiles. Results further show that the TIC's component responses remains uncompromised and degree of confidence in compound identification is improved.

1 Introduction

The use of gas chromatography coupled with mass spectrometer (GC/MS) for the evaluation of complex mixtures, such as those targeted by EPA Methods 8270 and 624 for the identification and quantitation of semivolatile and volatile organic compounds respectively, has been amply documented in the literature [1,2]. The procedure typically involves the generation of a total ion chromatogram (TIC). When the target compound levels are below the normal detection limits, the EPA allows separate selected ion monitoring (SIM), but the practice is expressly discouraged due to vastly reduced confidence in compound identification, except in cases where multiple ions are monitored for each compound. This paper describes a novel system that allows for the simultaneous acquisition of scan and SIMs. Since the data is acquired simultaneously, the uncertainty in compound identification is minimized and sample analysis time is reduced.

The E-MSD data acquisition system employs a combination of hardware, firmware and software to tune, calibrate and acquire data on single or quadrupole mass spectrophotometers. The data acquisition system allows the setup of several data collection methods or experiments for each sample injection. Thus, you can for instance acquire both a Scan and multi-ion SIM from the same run thereby eliminating injection port bias. A further benefit of the system is the availability of an enhanced electrometer option with an improved real dynamic range.

GC/MS chromatograms of semi-volatile mixtures were obtained using the normal scan mode of operation and the simultaneous scan/SIM mode. Chromatogram characteristics were examined to compare the two modes of operation.

2 Materials and Methods

The instrumentation employed in this study includes a Humble Instruments & Services, Inc. (HISI) Enhanced MSD (5970 Mass Spectrometer with an A.S.k ModCtrl board). Computer hardware and software consists of a PC with HP ChemStation for data processing and MStation hardware and software for the control of the 5970 (A product of Acquisition Solutions k). The GC was a standard HP 5890 equipped with a split splitless injection port. The column was a VB-5 (5% phenyl 95% methyl polysiloxane) 30m x 25mm x 25 μ m.

All standards were prepared in accordance with SW846 and EPA method 8270. Concentrates were purchased from Restek Corporation and Chem Service. Standards were prepared at levels of 0.5, 1.0, 2.0, 5.0 and 10.0 μ g/ml in dichloromethane. Samples were manually injected.

The chromatograms were obtained by injecting 2 μ l of the various semivolatile standards into the GC injection port. The GC conditions were as follows: Injector temp 280°C, splitless; oven initial temp 40°C for 2 minutes, followed by 6°C/min to 320°C.

3 Results and Discussion

The total ion chromatogram for a 1ppm semi-volatile standard mixture is shown in Fig.1.

EPA Method 8270 Semivolatiles Scan Mode

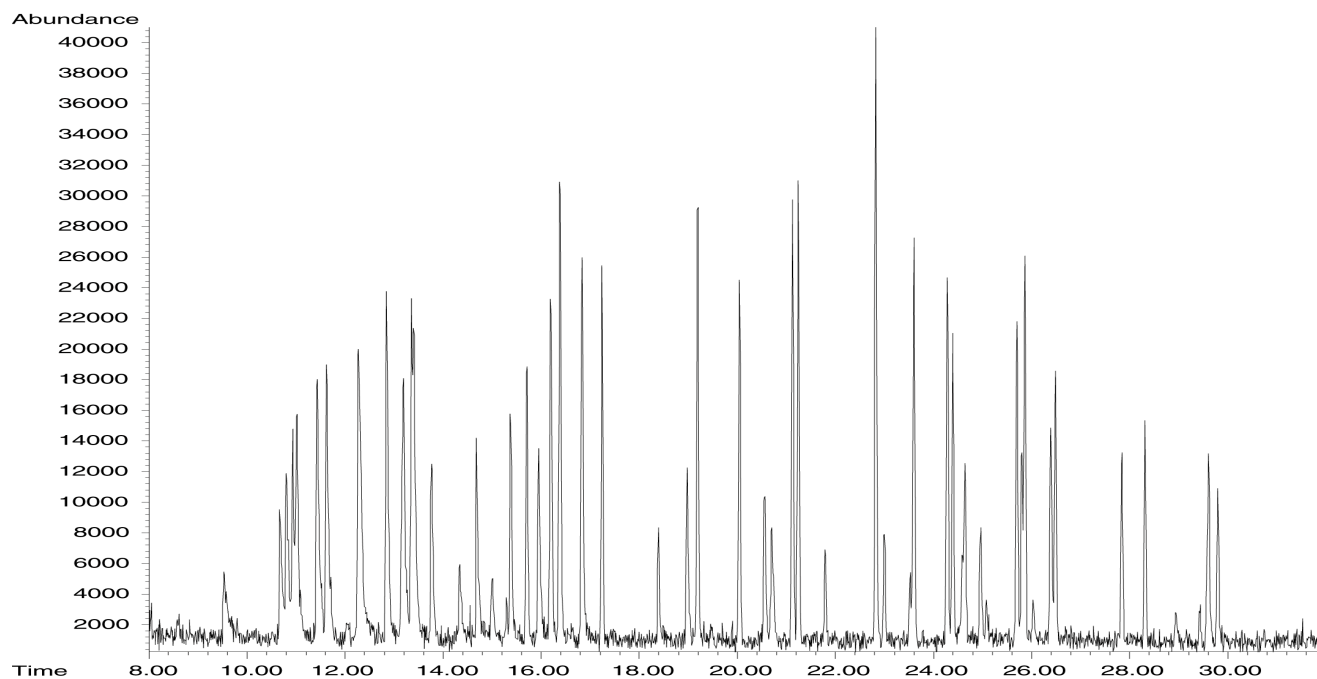


Figure 1. Total Ion Chromatogram of semi-volatile Mixture.

Selected slices of the total ion chromatograms run in the regular and simultaneous scan/SIM modes are compared in Fig. 2. Since peak heights and shapes are comparable in both modes of operation, method sensitivity is not compromised by the scan/SIM mode of operation even though fewer scans/per second are performed. Standard level is 0.5 ppm

Comparison of Total Ion Chromatograms for Scan and Simultaneous Scan/SIM Modes

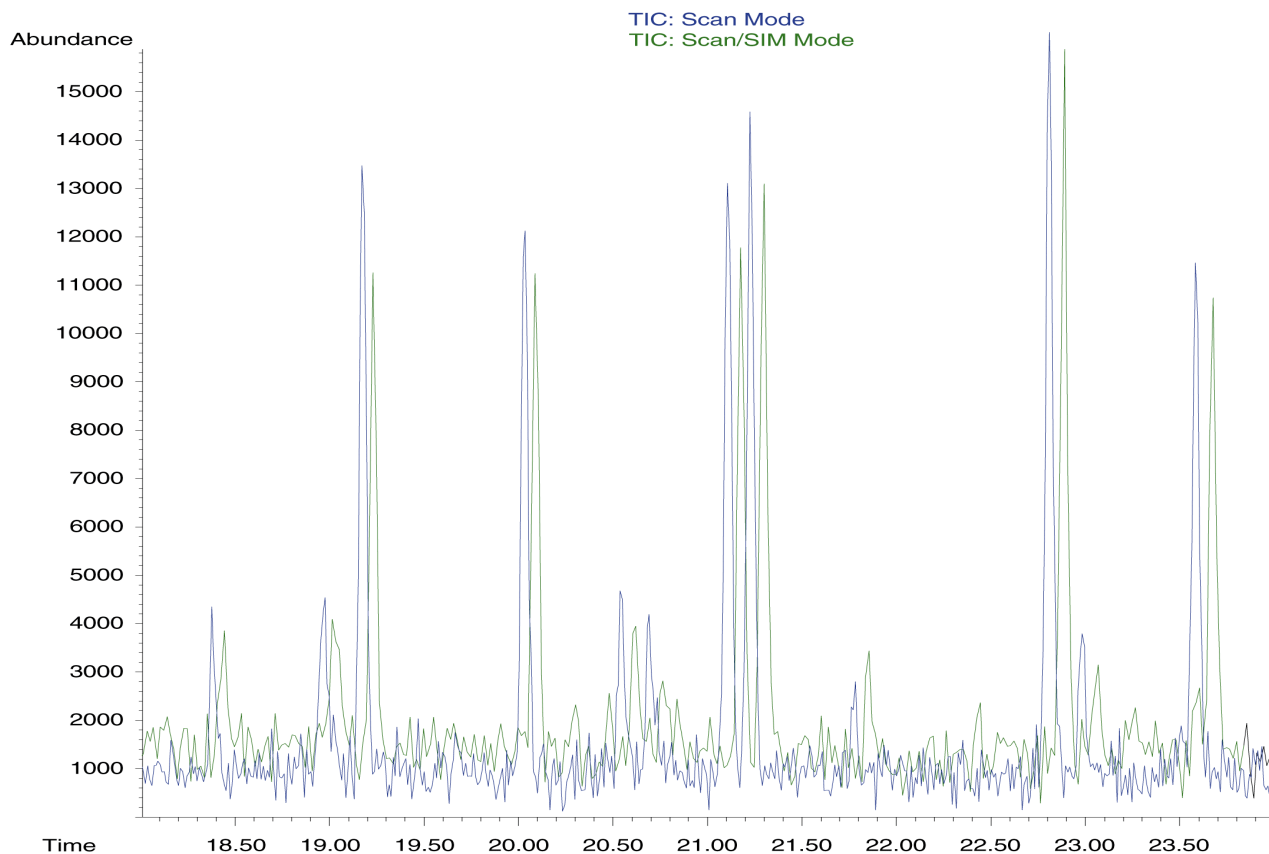


Figure 2

A comparison of the TIC obtained from the regular scan mode and a TIC of all targeted ions obtained in the SIM/scan mode (SIM component) of a 1.0 ppm standard is shown in Fig. 3. Targeted ions from the TICs shown in Fig. 3 are displayed in Fig. 4. In Fig. 3, the large peak at 23.7 minutes in the TIC run under scan mode is eliminated when the chromatogram is run under SIM mode, and only the targeted peak at 23.6 minutes remains. In addition, the signal to noise level is improved in the SIM/scan mode.

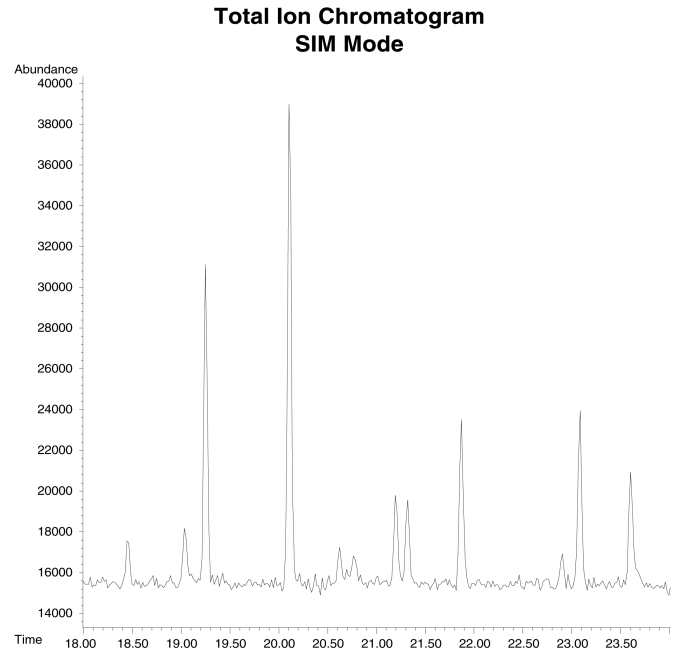
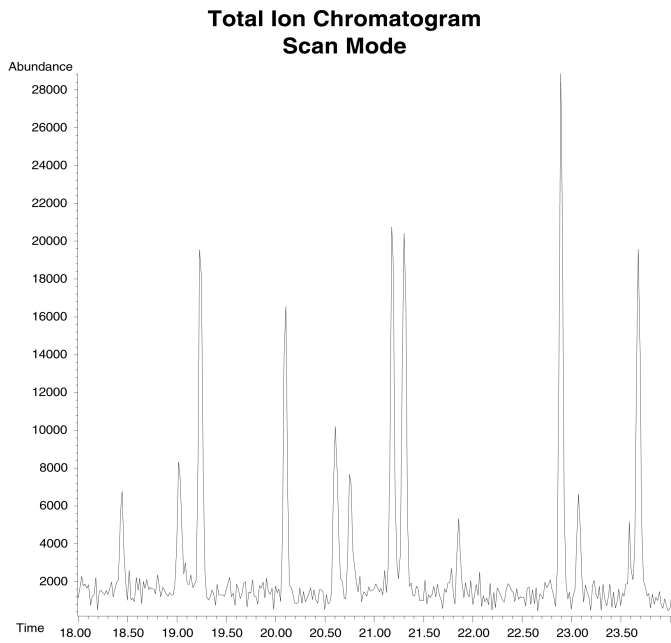


Figure 3

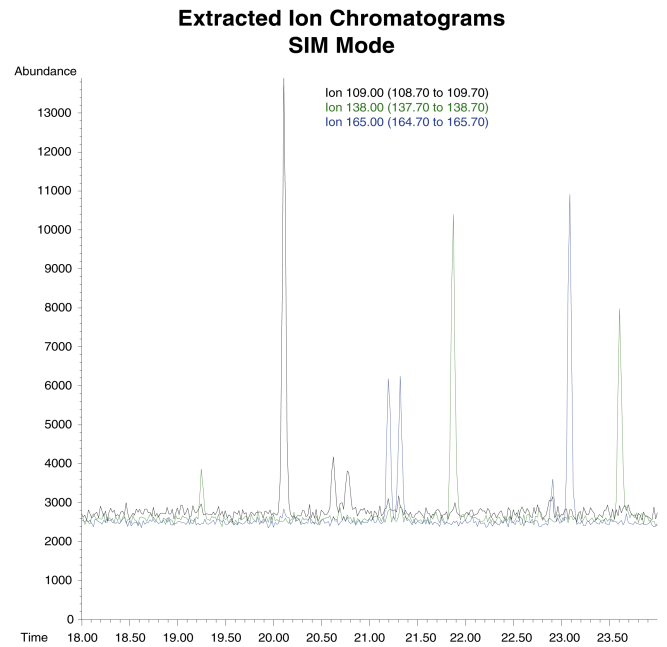
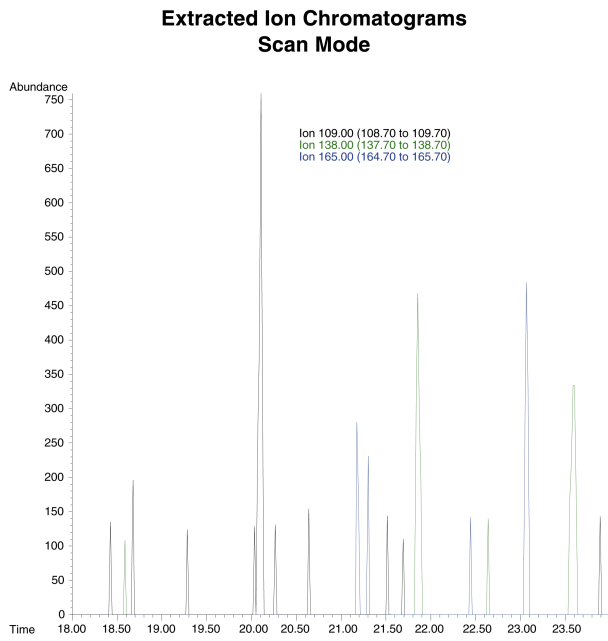


Figure 4

Comparison of m/z 138 Single Ion Chromatogram for Scan and SIM Modes

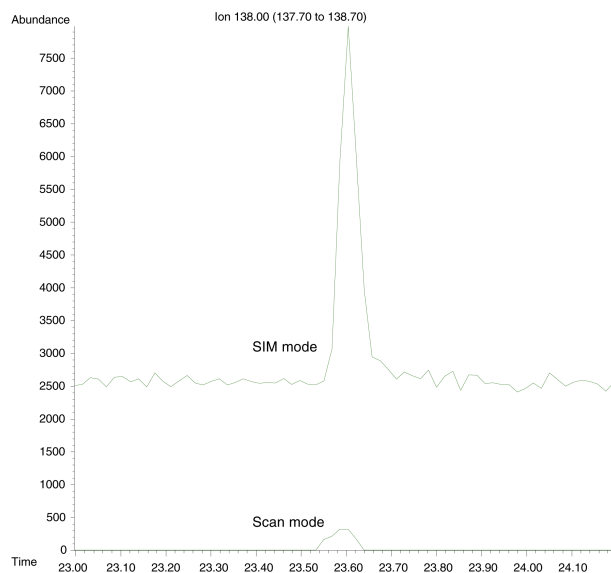


Figure 5

Conclusions

Despite the fact that operating the instrument in the multiple experiment mode lowered the number of scans/sec from 0.944 to 0.578 when targeting six ions in the SIM mode, there was no significant loss of sensitivity in the simultaneous scan/SIM run. Calibrations for both experiments were equally linear. In addition, the method detection limit for targeted problematic compounds was enhanced by a factor greater than ten.

The Combination of E-MSD software and exceptionally low bleed ValcoBond capillary columns can be employed to significantly improve limits of detection in various GC/MS analytical procedures while also improving the degree of confidence in acquired data.

5 Literature Cited

- [1] Eichelberger, J.W., Harris, L.E., and Budde, W.L, Anal. Chem. **1975**, *47*, 995-1000.
- [2] Engel, T.M., et al., "Screening of Semivolatile Organic Compounds for Extractability and Aqueous Stability by SW-846, Method 3510," Contract 68-03-3224, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH. June 5, 1987.

6 Acknowledgements

The authors would like to thank HISI Analytical Technologies (a division of Humble Instruments & Supplies, Inc. - www.humble-inc.com) for loan of the equipment used in the study.

E-MSD is a product of Acquisition Solutions k (A.S.k) . For information on E-MSD software and hardware upgrade go on the web at www.flash.net/~acqsol/