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Ion Trap - Different Problems and Discussion

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Ion Trap - Different Problems and Discussion

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Introduction

Finnigan introduced at spring 1995 a new ion-trap instrument on the market called GCQ. The instrument consist of an external ion source and an ion trap. The ion source is the same as the one used in the Finnigan SSQ quadrupole instrument. The GCQ could be used in different ionisation modes, like EI and CI, positive and negative ions. You can also setup the instrument in scan, SIM, and MS/MS mode. During a run you can mix the different scan modes. In MS/MS mode you can perform a full daughter ion scan of a parent ion or just follow a single daughter ion called Single Reaction Mode (SRM). The last technique should increase your sensitivity since you reduce the background. We have tested the instrument by analysing two steroid metabolites by using full scan, SIM and MS/MS mode.

Experimental

A solution containing two metabolites of Metandienone, namely 6- β -OH-metandienone and epimetandiol were analysed in full scan mode (fig 1 and 2) after derivatisation with MSTFA/NH₄I/ethanethiol 1000:2:3 (v:w:v) for 15 min at 60 C. From the full masspectrum a specific ion was selected to be used as the parent ion in the MS/MS experiment. The MS/MS scan of 6- β -OH-metandienone TMS-derivative is shown in fig. 3. The ion 517 was selected as parent ion since it was the most dominant ion in normal full scan mode. For the epimetandiol TMS-derivative we also used the most dominant ion 358 and MS/MS spectrum is shown in fig. 4

Two urine samples spiked with 50 ng 6 β -OH-metandienone and 50 ng epimetandiol respectively 5 ng 6- β -OH-metandienone and 5 ng epimetandiol was prepared using our normal method for analysing anabolic steroids. The samples were analysed in full scan, SIM and SRM mode.

The GC-column was a 17 meter ULTRA-1 from Hewlett-Packard with a film thickness of 0.11 μ m and an ID of 0.2 mm. Column head pressure was set to 8.8 psi with a splitter of 1:15. Temperature program: 180 C, 3 C per min., 231 C, 30 per min, 320 C, 2 min. 3 μ l of the derivatisation solution was injected to the GCMS.

In SRM mode a collision energy of 1.5 was used with 1 second per scan.

Results

The S/N ratio were calculated for the two steroid peaks. The results are shown in the following table,

6 β -OH-metandienone

	SCAN 50-650 m/z	SRM 517 -> 229	SIM 517
25 ng/ml sample	29.2	542.7	17.5
2.5 ng/ml sample	4.6	38.4	8.9

Epimetendiol

	SCAN 50-650 m/z	SRM 358-> 301	SIM 358
25 ng/ml sample	14.3	82.2	12.8
2.5 ng/ml sample	not detected	not detected	not detected

The best results show the measurement of 6 β -OH-metandienone. This is probably due to a dominant base peak 517 compared to the 358 from epimetendiol. Several more experiments have to be performed to fully investigate the function of the GCQ. There are several parameters that one can change and it is more complicated to understand how the instrument behave compared to a common benchtop quadrupole instrument. The software for GCQ is very simple regarding the possibility to automatically produce your own report of chromatographic data. This has been a limiting step during the testing of the instrument. For the moment the instrument is more a research grade massspectrometer than a system used for routine application.

Spectrum Plot C:\GCQ\DATA\EXP9602\MDNMET02 01/24/96 12:52:21
 Comment: 6b-OH-metandienone TMS 200 ng total
 Scan No: 815 Retention Time: 16:37 RIC: 1241590 Mass Range: 51 - 642
 # Peaks: 448 Base Pk: 517 Ioniz: 672 us Int: 188930 100.00% = 188930

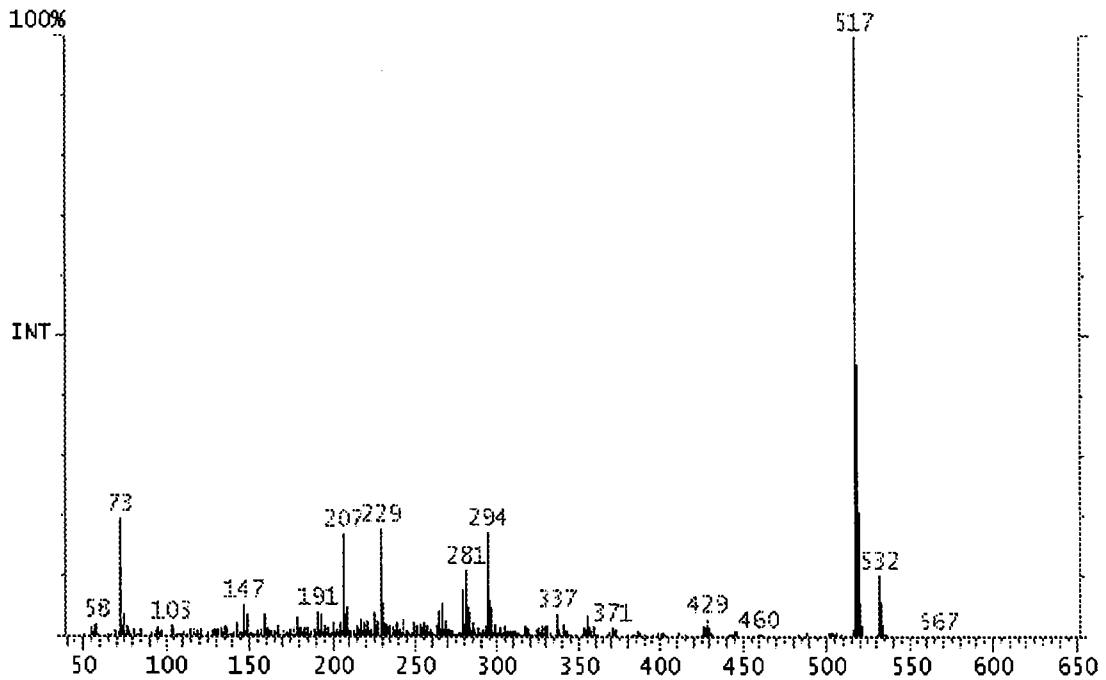


Fig. 1: Mass spectrum of 6b-OH-Metandione-bis-TMS. Molecular ion 532 and base peak 517 (M-15).

Spectrum Plot C:\GCQ\DATA\EXP9602\MDNMET04 01/24/96 13:51:53
 Comment: Epimetendiol (metabolit IV) TMS 200 ng total
 Scan No: 381 Retention Time: 9:23 RIC: 1061988 Mass Range: 51 - 649
 # Peaks: 407 Base Pk: 358 Ioniz: 762 us Int: 61284 100.00% = 61284

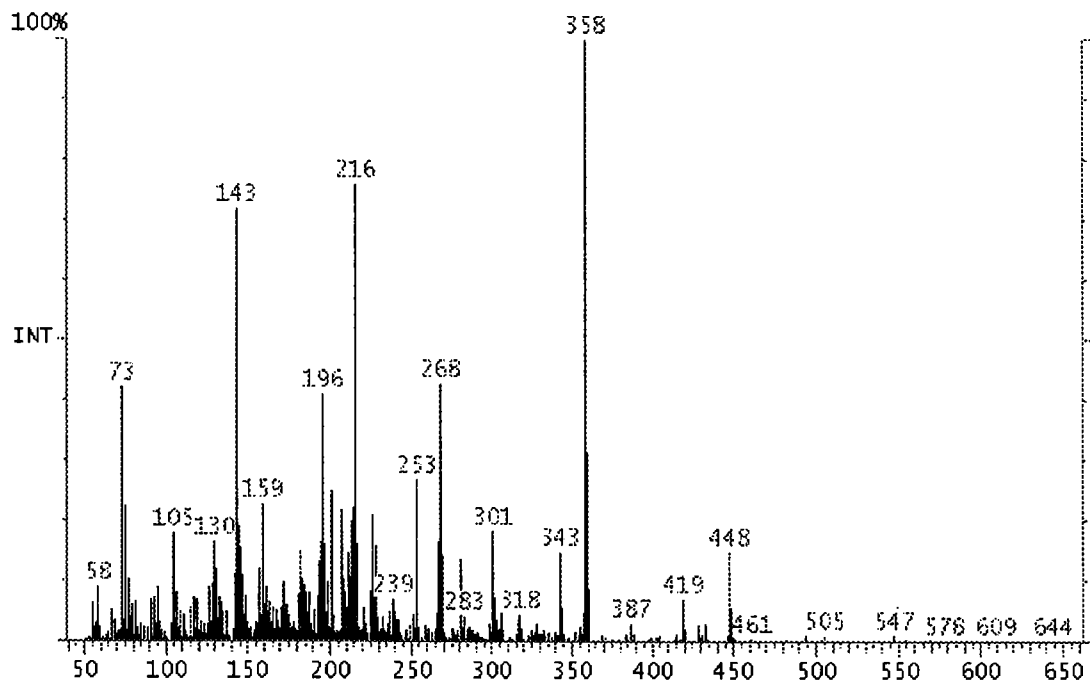


Fig. 2: Mass spectrum of epimetendiol-bis-TMS. Molecular ion 448 and base peak 358 (M-90).

Spectrum Plot C:\GCQ\DATA\EXP9602\MDNMET09 01/11/96 18:14:14
 Comment: 6b-OH-MDN MS/MS 517 (1.5) scan 150-550
 Scan No: 821 Retention Time: 16:43 RIC: 106471 Mass Range: 150 - 544
 # Peaks: 259 Base Pk: 229 Ioniz: 4722 us Int: 18189 100.00% = 18189

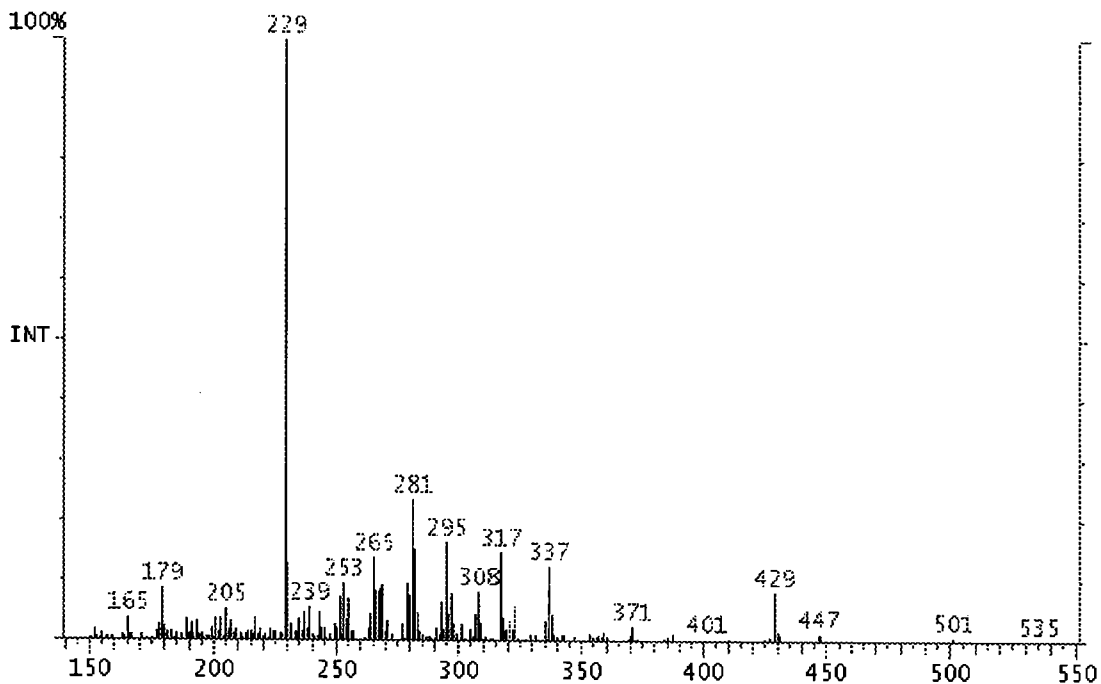


Fig. 3: MS/MS spectrum of ion 517, 6b-OH-Metandione-bis-TMS.

Spectrum Plot C:\GCQ\DATA\EXP9602\MD-MSMS4 01/25/96 12:16:18
 Comment: Epimetendiol TMS 200 ng total ms/ms scan 100:360 col 1.5
 Scan No: 381 Retention Time: 9:23 RIC: 42188 Mass Range: 103 - 358
 # Peaks: 180 Base Pk: 301 Ioniz: 24584 us Int: 20418 100.00% = 20418

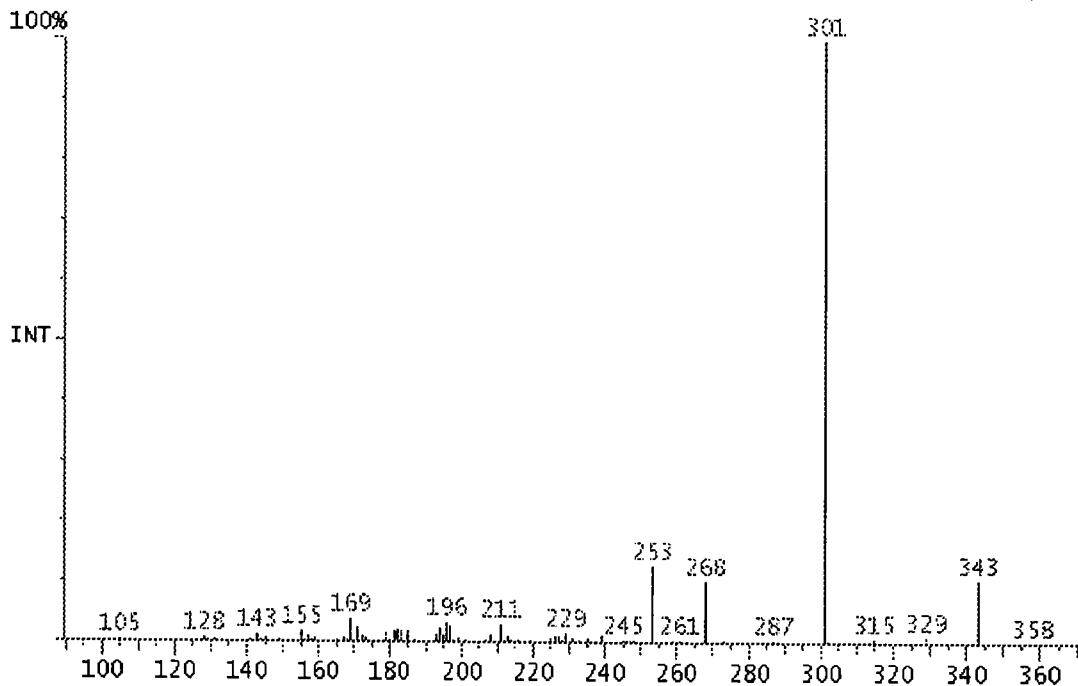


Fig. 4: MS/MS spectrum of ion 358, epimetendiol-bis-TMS