



1.7 Analysis of Pharmaceuticals Using Direct Sample Inlet Method, EI, CI and Mass Spectrometry (1) - GCMS

•Explanation

Composite samples are difficult to analyze with MS using direct inlet (DI) because GC separation is not used, but direct inlet MS is effective in analysis of compounds easily heat decomposed or compounds with high boiling points. If the compound has been purified in advance, the structure can be confirmed quickly without having to make a derivative of it.

The structure and molecular weight of such a compound can be confirmed using a combination of electron-impact ionization (EI) and chemical ionization (CI) methods.

Maleic acid ergometrine is maleate of ergometrine. As shown in Fig. 1.7.7 to 1.7.9 and Fig. 1.7.10 to 1.7.12, the **salt is decomposed by heating**, ergometrine and maleic acid appear, and can be separated for observation using the differences in boiling points after decomposition.

•Analytical Conditions

Instrument	:GCMS-QP5050A (with CI/DI)
— MS —	
Interface Temp.	:250 °C
Ionization Method	:EI/CI (reaction gas: isobutane)
Scan Range	:m/z 35-700 (EI) m/z 100-700 (CI)
Scan Interval	:2sec
DI Temp.	:Room temperature - 10°C/min-350°C (10 min) (sample 1, 2, 3) Room temperature - 20°C/min-350°C (10 min) (sample 3 and 4)

Chart 1.7.1 Sample List

No.	Pharmaceuticals		Molecular Weight	Application
1	Japanese Pharmacopoeia standard product	Prednisolone	360	Adrenocorticotropic hormone action for full and local use
2	Japanese Pharmacopoeia standard product	Reserpine	608	Antipsychotic agents, antihypertensives, sympathetic neuron blockers
3	Japanese Pharmacopoeia standard product	Maleic acid ergometrine	441	Uterotonics, hemostatic drugs
4	Commercially available	Indomethacin externally applied medicine	357	Antipyretic painkiller, antiphlogistic drugs

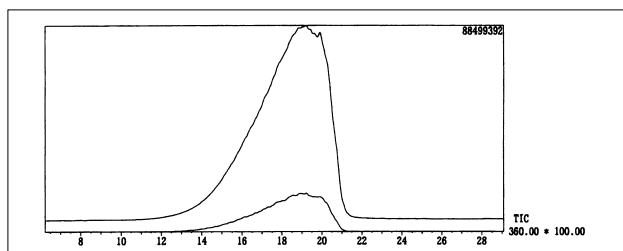


Fig. 1.7.1 Total ion chromatogram of prednisolone

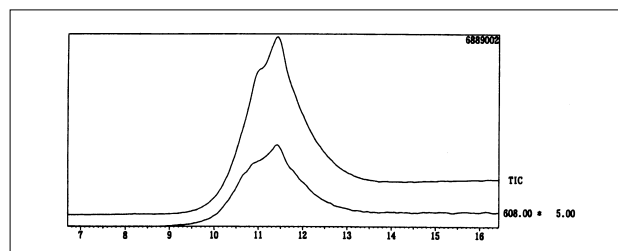


Fig. 1.7.4 Total ion chromatogram of reserpine

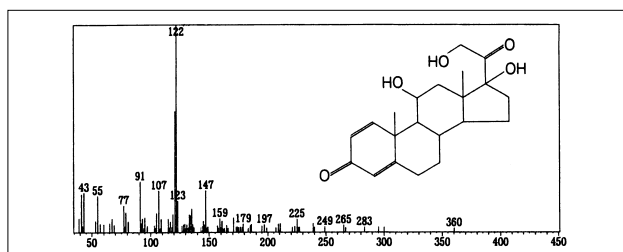


Fig. 1.7.2 EI mass spectrum of prednisolone

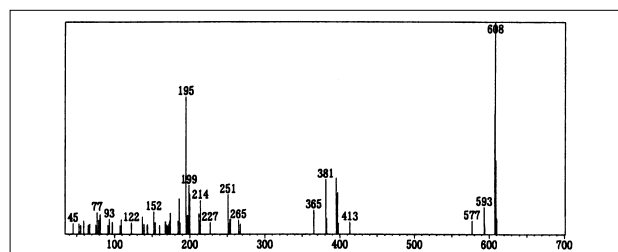


Fig. 1.7.5 EI mass spectrum of reserpine

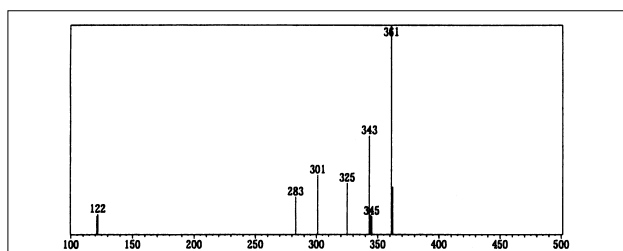


Fig. 1.7.3 CI mass spectrum of prednisolone

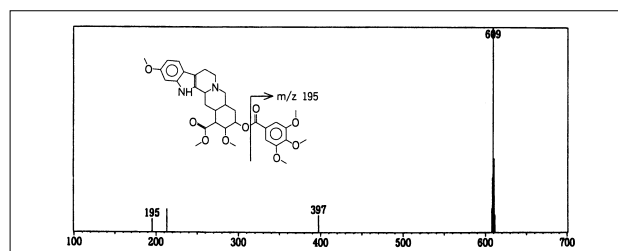


Fig. 1.7.6 CI mass spectrum of reserpine

1.7 Analysis of Pharmaceuticals Using Direct Sample Inlet Method, EI, CI and Mass Spectrometry (2) - GCMS

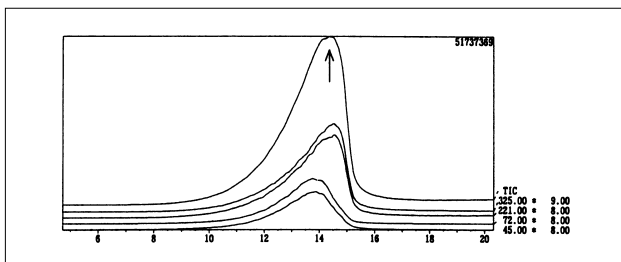


Fig. 1.7.7 Total ion chromatogram of maleic acid ergometrine using EI

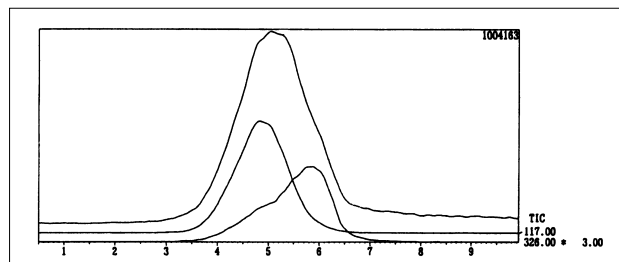


Fig. 1.7.10 Total ion chromatogram of maleic acid ergometrine using CI

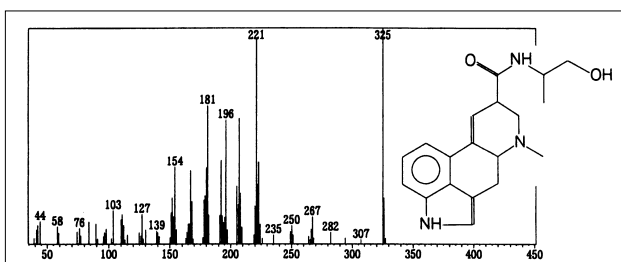


Fig. 1.7.8 EI mass spectrum of ergometrine

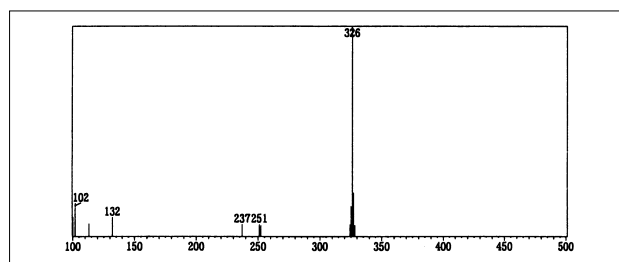


Fig. 1.7.11 CI mass spectrum of ergometrine

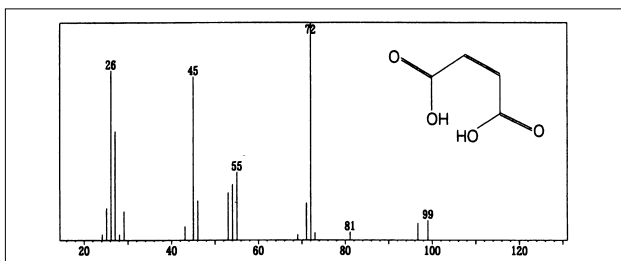


Fig. 1.7.9 EI mass spectrum of maleic acid

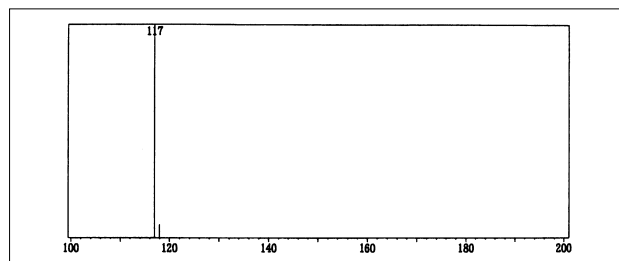


Fig. 1.7.12 CI mass spectrum of maleic acid

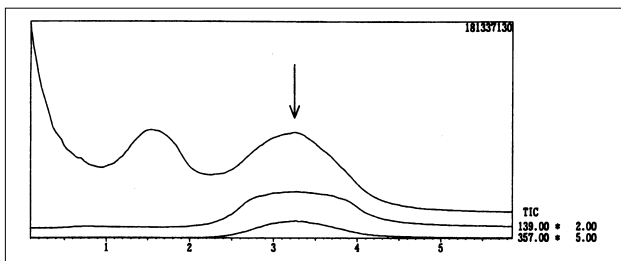


Fig. 1.7.13 Total ion chromatogram of indomethacin externally applied medicine

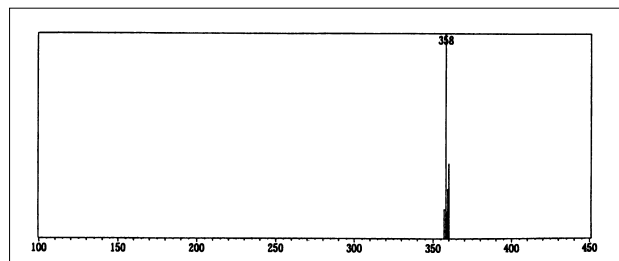


Fig. 1.7.15 CI mass spectrum of indomethacin

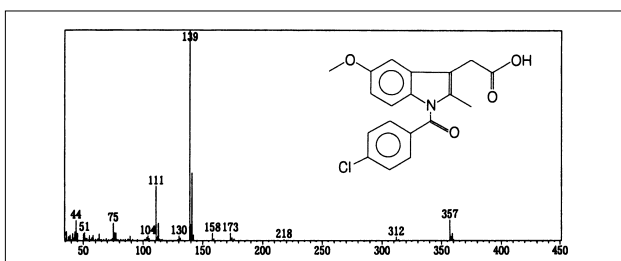


Fig. 1.7.14 EI mass spectrum of indomethacin