



3.4 NCI-GC/MS Analysis of a Crude Drug (Red Ginseng) - GCMS

•Explanation

The items of total BHC (α , β , γ , δ -BHC) and total DDT (DDT, DDE, DDD) have been newly added to the purity testing method for crude drugs. Here, the GC-ECD measuring method is used because these items are chlorine-group agrochemicals. The negative chemical ionization method (NCI-GC/MS) shows the same high sensitivity and selectivity as the ECD method for compounds with electron affinity. The NCI-GC/MS method was used to analyze targeted agrochemicals in crude drugs.

•Pretreatment

The following shows the pretreatment method.

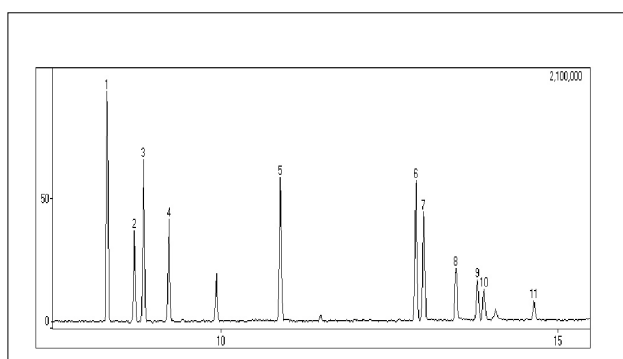
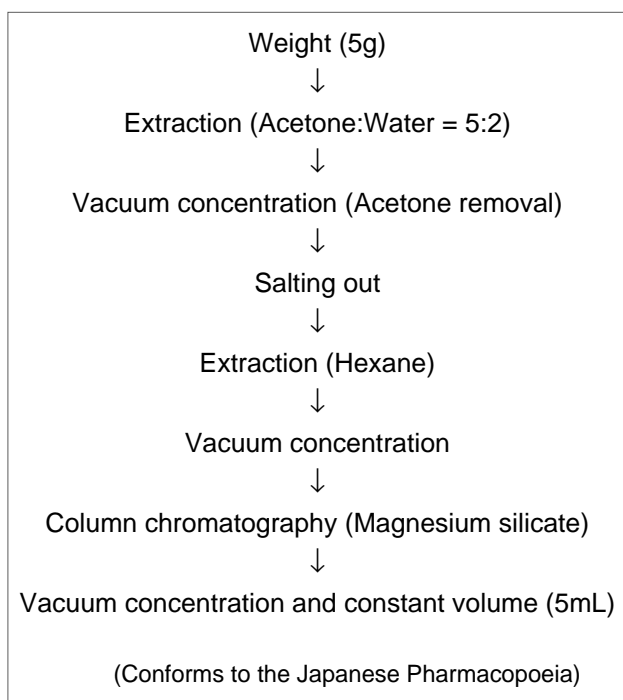


Fig. 3.4.1 Total ion chromatogram of analyzed crude drug (red ginseng)

•Analytical Conditions

Instrument	: GCMS-QP5050A
— GC —	
Column	: DB-5MS (30m × 0.25mm.i.d. df=0.25 μ m)
Column Temp.	: 50°C (2min) - 30°C/min - 180°C - 8°C/min - 280°C (3min)
Injector Temp.	: 250°C
Carrier gas	: He 100kPa
Injection Method	: Splitless (Sampling Time=2min)
Injection Volume	: 2 μ L
— MS —	
Interface Temp.	: 260°C
Ionization Method	: NCI(CH ₄ 3.5kgf/cm ²)
Scan Range	: m/z 35-350
Scan Interval	: 0.5sec

Chart 3.4.1 Targeted Agrochemicals

Peak No	Name	Peak No	Name
1	α -BHC	7	Dieldrin
2	β -BHC	8	Endrin
3	γ -BHC	9	p,p'-DDD
4	δ -BHC	10	o,p'-DDT
5	Ardrin	11	p,p'-DDT
6	p,p'-DDE		

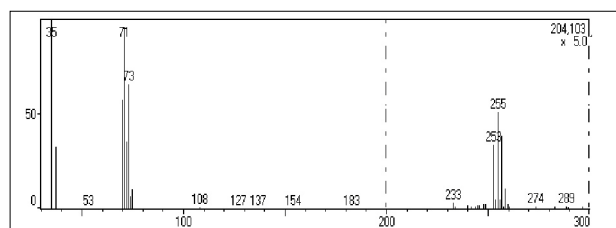


Fig. 3.4.2 NCI mass spectrum of γ -BHC (peak-3)

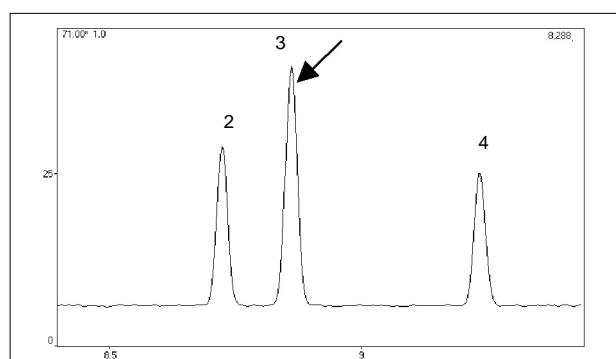


Fig. 3.4.3 SIM chromatogram (NCI) of γ -BHC (m/z:71)