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Presence of α-Pyrrolidone in Ginseng Extracts.

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禹麟根・徐廷祥・張正子・申國鉉：人蔘 "methanolic extract"中의 "α-pyrrolidone" 證明

人蔘의 "methanolic extract"을 分劃하여 鹽基性 成分을 모으고 GLC-Mass on line, NMR spectrum에 의하여 α-pyrrolidone과 數種의 鹽基性物質이 共存함을 證明하였다.

Introduction

In earlier investigation, it was reported by one of us that a fraction of ginseng methanolic extract suppressed the growth rate of HeLa and KB cells a concentration of 20r/ml of cultural media[1]. We proved α-pyrrolidone previously unreported in the above fraction by gas liquid chromatography- mass spectrometry on line and nuclear magnetic resonance spectrometry. (cf. Fig. 1.)

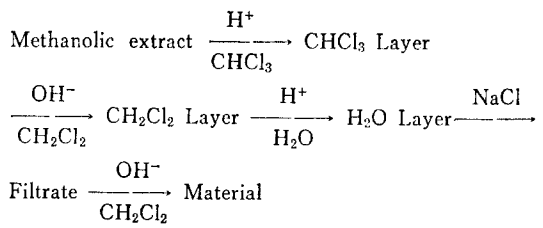


Fig. 1. Scheme of fractionation for the material

In appling of the material into mass spectrometer at inonization voltage 70 eV the mass spectra showed no sigificant fragmentation pattern to check of molecular ion, however, by lowering of ionization voltage to 18 eV several enhanced peaks were able to be observed. The resulting peaks seemed to be molecular peaks and their milli-mass were measured by photoplate method (cf. Fig 2 and Table I).

After purification of the material by preparative gas liquid chromatography mass spectra of the first band just after solvent peak was taken on both the left and right side on the band (cf. Fig. 3).

Condition: Column, 3% SE-30 on chromosorb G, Column temperature programing at 5/min

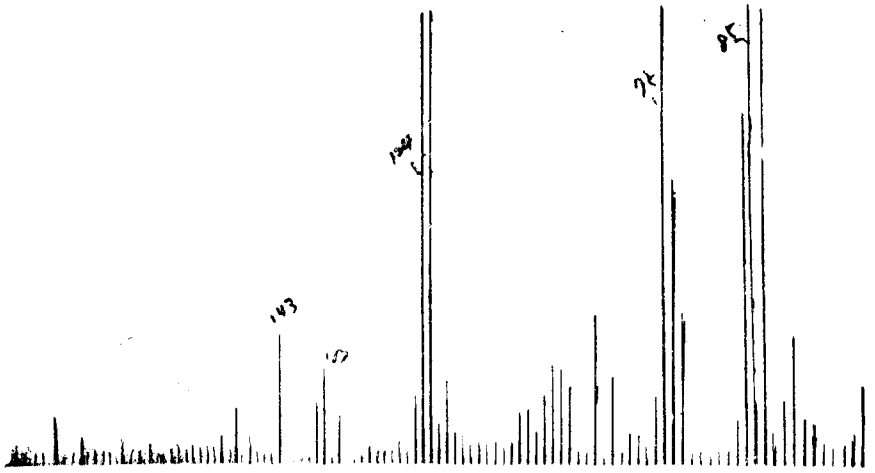


Fig. 2. Mass spectrum of the material at 18 eV.

Table I. Milli-mass observed on the spectrum the material and calculated value.

PARENT OR FRAGMENTATION m/e	MILLIMASS		
	OBSVD.	CALCD.	FORMULA
143 (C_9H_5NO)	0.035	0.036	$C_7H_3N_4$
		0.037	C_9H_5NO
		0.033	$C_4H_5N_3O_3$
		0.034	$C_6H_7O_4$
137 ($C_6H_7N_3O$)	0.062	0.059	$C_6H_7N_3O$
		0.060	$C_8H_9O_2$
124 ($C_6H_8N_2O$)	0.063	0.062	$C_4H_6N_5$
		0.064	$C_6H_8N_2O$
95 ($C_5H_7N_2$)	0.061	0.061	$C_5H_7N_2$
85 (C_4H_7NO)	0.055	0.053	C_4H_7NO

from 170°—280°C, TCD. temperature 330°C, injector port 350°C, He flow rate 40 ml/min, mass analysis at 70 eV. Mass spectral analysis of the purified material gave a value of m/e 85 for the molecular weight which agrees with formular C_4H_7ON determined from mill-mass (cf. Table I). Further breakdown into m/e 56, 42, and 30 would explain respectively as below. If the instense peak at m/e 30 came from $-CH_2-NH+H$(rearrangement) and one of unsaturation degree for the formular, C_4H_7ON, is due to carbonyl bond, the structure can be postulated to be α- or β-pyrrolidone.

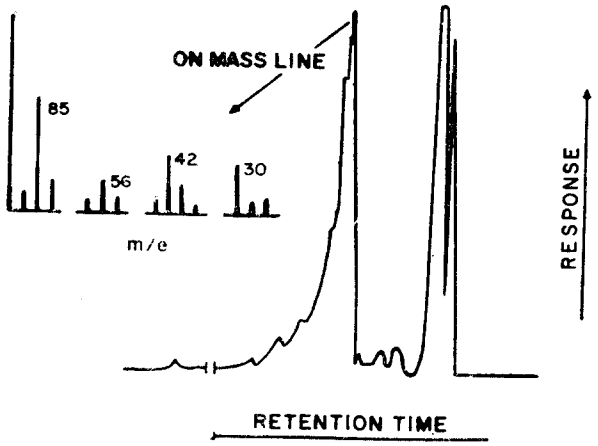
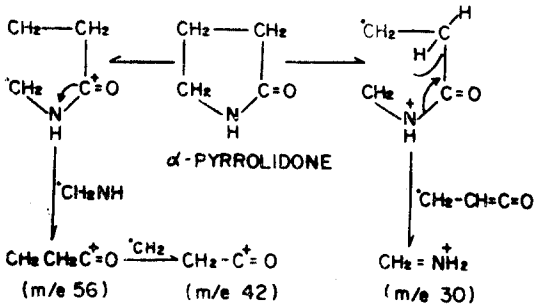


Fig. 3. Gas liquid chromatography and mass spectra of the material.



For the final confirmation, the first GLC band, which was applied into mass spectrometer, was preparatively collected and the NMR spectra of the collected liquid in $CDCl_3$ at 23°C provided evidence for the presence of α-pyrrolidone in the material. The assignment of multiple peaks in the region τ-2–3.5 ppm due to -CH_2-CH_2-CH_2-CO- and broad peak at 6.5–7.5 ppm due to -NH was agreeable with that of reference spectra of α–pyrrolidone[2] (cf. Fig. 4).

Key: O, responsible peak upon α-pyrrolidone; X due to impurity. We present here that several basic components are associated with α-pyrrolidone in the material which was found to

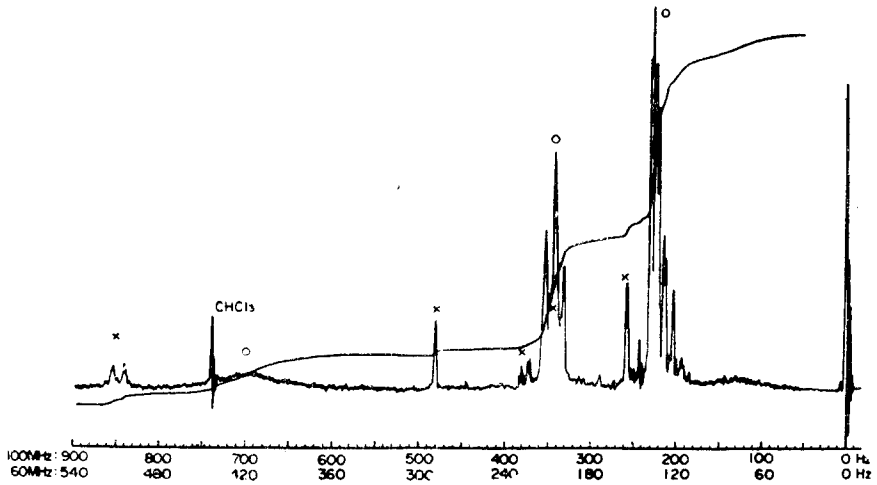


Fig. 4. NMR-Spectrum of the first GLC band which was applied into mass spectrometer.

suppress HeLa and KB cells growth rates.

Experimental

Gas chromatography-Mass spectrometry. -Gas chromatography condition are shown in the figure 3. The combined gas chromatography-mass spectrometer was fitted with 7ftx1/8 in. column containing 3%SE-30 on 100-120 mesh of chromosrob G, scan time were 4-5sec.; ionization potential was 70-18 eV. NMR spectrum of the fraction after preparatively purified by GLC was taken in $CDCl_3$ at 23°C.

References

1. Lin Keun Woo, et al., Ital. patol. Clin. Tumor., 9, 53-61 (1965).
2. Varian. NMR -spectra No. 68 (1962).