CHAPTER IV

DISCUSSION

The dried coarsely powdered barks (4 kg.) was macerated with methanol. The chloroform extract was then partitioned according to the process shown in previous chapter. Three compounds, codename AM-1, AM-3 and TS-1 were isolated from the chloroform fraction.

1. Structure Elucidation of AM-1

AM-1 was white needle crystals. The mass spectrum of AM-1 (Figure 10) revealed the molecular ion peak ($M^+ + 1$) at *m/z* 310 (100 %) which corresponded to the molecular formula of $C_{17}H_{31}N_3O_2$. The spectral data were in agreement with those obtained previously for palustrine (Natsume, Ogawa and Motoc, 1984) and their identity was confirmed by comparison (^{1}H-NMR, MS) with authentic data. The ^{1}H-NMR spectra (Figure 11-12), which have a doublet peak of 6.86 ppm indicated that it was an amide. Two triplet peaks at 5.73 and 5.63 ppm indicated the presence of olefin. The broad singlet peak at 4.65 ppm indicated a hydroxyl group, attached to C-18. The structure of alkaloid AM_1 was confirmed by its mass spectrum that it showed four characteristic fragmentation (Figure 7). The molecular ion (M^+), m/e 309 was eleminated by α-splitting, $M^+ - CH_2CH_3$ into m/e 280 (4%); $M^+ - CH_2CH_3 - CH_2O$ into m/e 250 (7%).

Table 1 ^{1}H-NMR ($CDCl_3$ / TMS)

Chemical shift (δ , ppm)	Position (Proton)	Multiplicity	Coupling constant (J, Hz)
3.88	5-NH (amine)	d	15
4.12	18 (H)	m	
4.65	18 (OH)	br,s	
5.63	15 (H)	t	10
5.73	14 (H)	t	10
6.83	10-NH (amide)	d	10

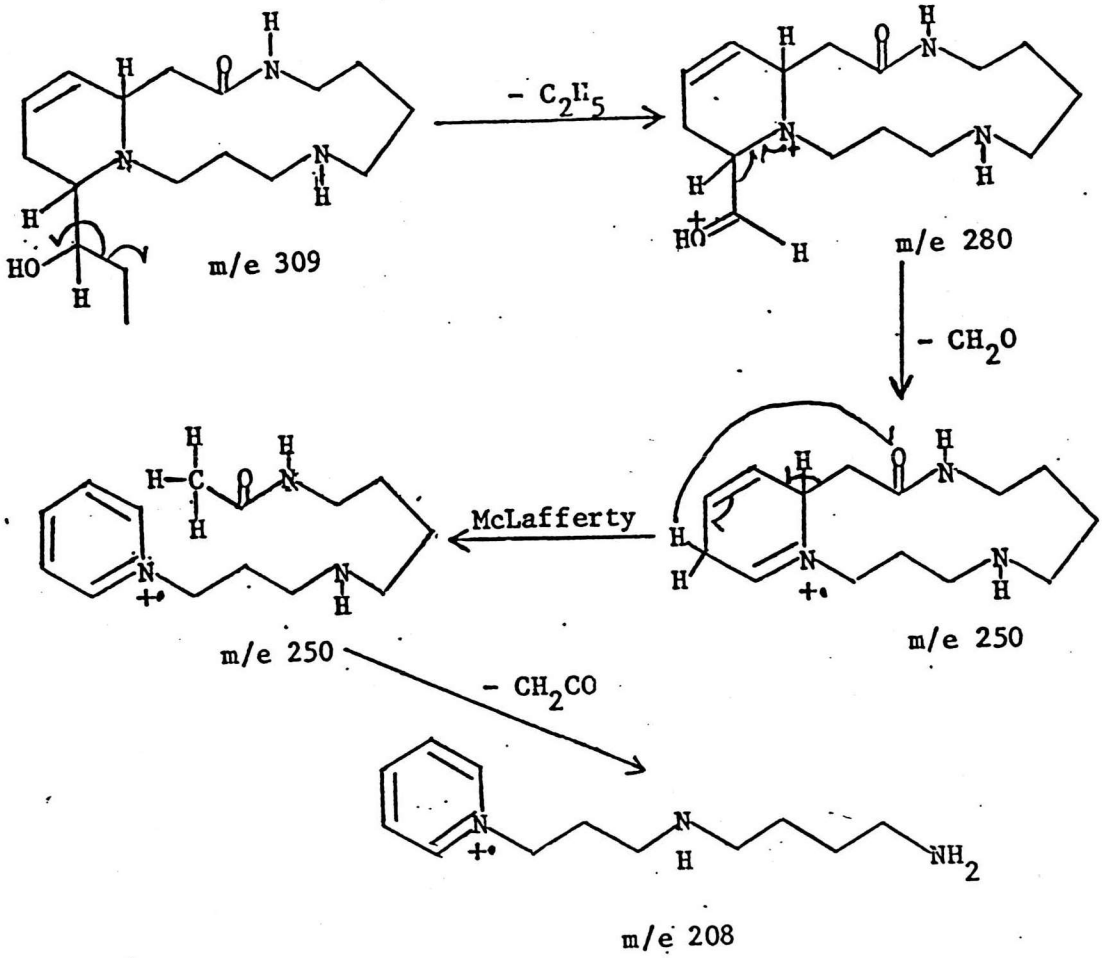
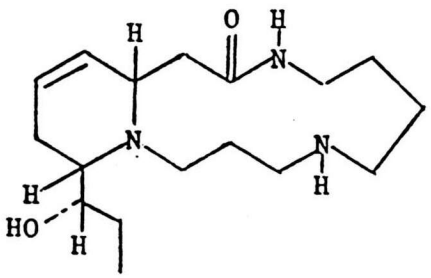


Figure 7 Fragmentation of Palustrine

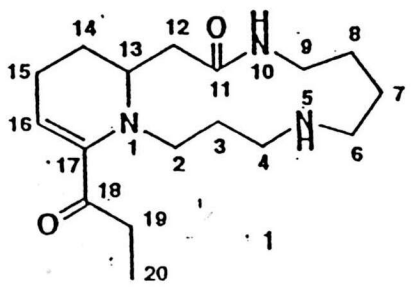
From spectral data (MS, NMR, ^{13}C NMR) iddicated that this compound was palustrine as showed below.



Palustrine

2. Structure Elucidation of AM-3

AM-3 was yellow oil mass. The mass spectrum of AM-3 (Figure 13) revealed the molecular ion peak (M^+ + 1) at *m/z* 308 (100 %) which corresponded to the molecular formula of $C_{17}H_{29}N_3O_2$. The spectral data were in agreement with those obtained previously for albizzine A (Ito *et al* , 1994) and their identity was confirmed by comparison (^{1}H-NMR, MS) with authentic data. The ^{1}H-NMR spectra (Figure 14-15), show the olefenic proton at 6.75 ppm as triplet.This spectral data did not show the hydroxyl group at about 4.58 ppm. From the spectral data (MS, NMR) indicated that this compund was albizzine A as showed below.



Albizzine A

3. Structure Elucidation of TS-1

TS-1 was obtained as white needle crystals. The mass spectrum of TS-1 (Figure 16) revealed the molecular ion peak at *m/z* 454 (7%) and accurate mass was consistent with the molecular formula $C_{30}H_{46}O_3$

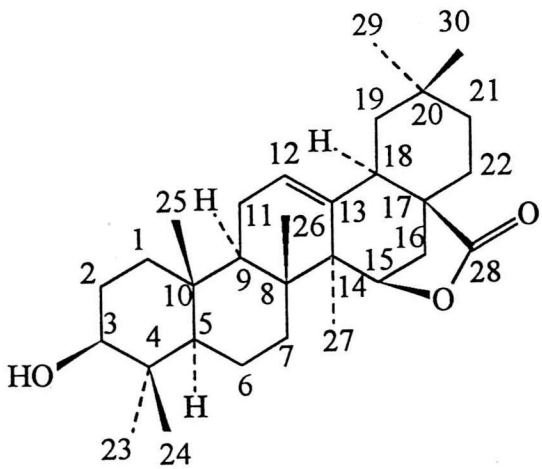
TS-1 could be assigned as a known pentacyclic triterpene, triptotripterpenoidallactone A (*Zhang et al*, 1993) by the analysis of its ^{1}H and ^{13}C-NMR spectra. The ^{1}H-NMR spectra (Figure 17-19) showed it signals at δ 0.7 - 1.2 ppm. which were signals of 7 methyl proton. The signals at δ 1.2 - 2.4 ppm. were the signals of methylene and methine protons. The signal at δ 3.38 ppm (m) was the signal of the proton at C-15. The signal at δ 4.18 ppm (dd, J = 6 Hz) was the signal of the proton at C-3 proton. The olefinic signal at δ 5.31 ppm. (t, J = 3 Hz) could be assigned to C-12 and sharp signal at δ 7.3 ppm (s) could be assigned to hydroxyl proton.

Analysis of the ^{13}C-NMR and DEPT spectra of TS-1 (Figure 20-24) revealed the presence of 9 methylene carbon (δ 18.1 ppm , δ 23.3 ppm , δ 24.2 ppm , δ 25.0 ppm , δ 26.6 ppm , δ 32.9 ppm , δ 33.2 ppm , δ 38.4 ppm , δ 39.6 ppm), 6 methine carbons (δ 43.1 ppm , δ 47.3 ppm , δ 55.0 ppm , δ 78.5 ppm , δ 83.3 ppm , δ 124.6 ppm), 7 methyl carbon (δ 15.4 ppm , δ 15.4 ppm , δ 16.7 ppm , δ 20.6 ppm , δ 23.7 ppm δ 24.7 ppm , δ 27.8 ppm), 8 quaternary carbon (δ 35.0 ppm , δ 36.85 ppm , δ 38.5 ppm , δ 39.1 ppm , δ 39.5 ppm δ 42.3 ppm , δ 139.9 ppm , δ 182.9 ppm) including one carbonyl carbon.

Table 2 ^{13}C NMR-Chemical shifts (δ) ppm of TS-1

Carbon position	TS-1	Carbon position	TS-1
C-1	33.2	C-16	24.2
C-2	25.0	C-17	42.3
C-3	83.3	C-17	43.1
C-4	39.1	C-19	39.6
C-5	55.0	C-20	35.0
C-6	18.1	C-21	38.4
C-7	32.9	C-22	26.6
C-8	36.85	C-23	16.7
C-9	47.3	C-24	20.6
C-10	38.5	C-25	23.7
C-11	23.3	C-26	15.4
C-12	124.6	C-27	15.4
C-13	139.9	C-28	182.9
C-14	39.5	C-29	24.7
C-15	78.5	C-30	27.8

Form these spectral data (MS, ^{1}H NMR, ^{13}C NMR) indicate that this compound was triptotripterpenoidallactone A as shown below.



Triptotripterpenoidallactone A