

CHAPTER 2

INVESTIGATION ON THE ACID PART OF FLACOURTIA SEPIARIA

Chromatography of the acid part

The acid part of the benzene extract of powdered Flacourtia sepiaria was chromatographed over silica gel column and the following fractions were collected (Table 6).

Table 6

Chromatography results of the acid part of F sepiaria

Fraction number	Eluent	Nature of residue after removal of solvent	Melting point
1	Petroleum ether	waxy solid	80- 82°
2	Pet ether:benzene (4:1)	oil	-
3	Pet ether:benzene (3:2)	solid	117-120°

Further elution with more polar solvents did not afford any solid materials.

Examination of fraction 1 (Table 6) : isolation and identification of tetracosanoic acid

Fraction 1 was purified by crystallization from methanol to give waxy solid, mp 86-87° and was analysed for $C_{24}H_{48}O_2$.

The IR spectrum (Fig 23) of the compound shows absorption band at 1700 cm^{-1} for a carbonyl function, which is disappeared when the compound was esterified with diazomethane and the new bands at 1735 and 1170 cm^{-1} , characteristic for methyl ester, appear in the IR spectrum (Fig 24).

The ^1H NMR spectrum (Fig 25) of the methyl ester shows peaks at 3.65 (s), 2.30 (t), 1.62 (m), 1.60 (broad) and 0.90 (t) ppm. The singlet at 3.65 ppm is due to the ester methyl and the triplet at 0.90 ppm with $J = 8\text{ Hz}$ is due to a primary methyl group. The triplet at 2.30 and a multiplet at 1.62 ppm, each integrated for two protons, indicate two alpha and two beta protons with respect to the carbonyl carbon of the ester, which are spin-spin coupled to each other with $J = 8\text{ Hz}$. The broad band centred at 1.60 ppm is integrated for 40 protons which are accountable for 20 CH_2 groups in a saturated acyclic system attached to $-\text{CH}_2-\text{CH}_2-\text{COOCH}_3$ moiety. Therefore, the ester is methyltetracosanate and the acid found in fraction 1 (Table 6) is tetracosanoic acid. The ^{13}C NMR (Fig 26) of the ester is also consistent with methyltetracosanate. The ester carbonyl carbon appears at 174.4 ppm. The peak at 51.2 ppm is due to the methyl carbon of the ester group. The band around 30 ppm is integrated for 18 carbon atoms.

However, the melting points and optical rotations of tetracosanoic acid and its methyl ester were found to be similar with reported values¹⁰.

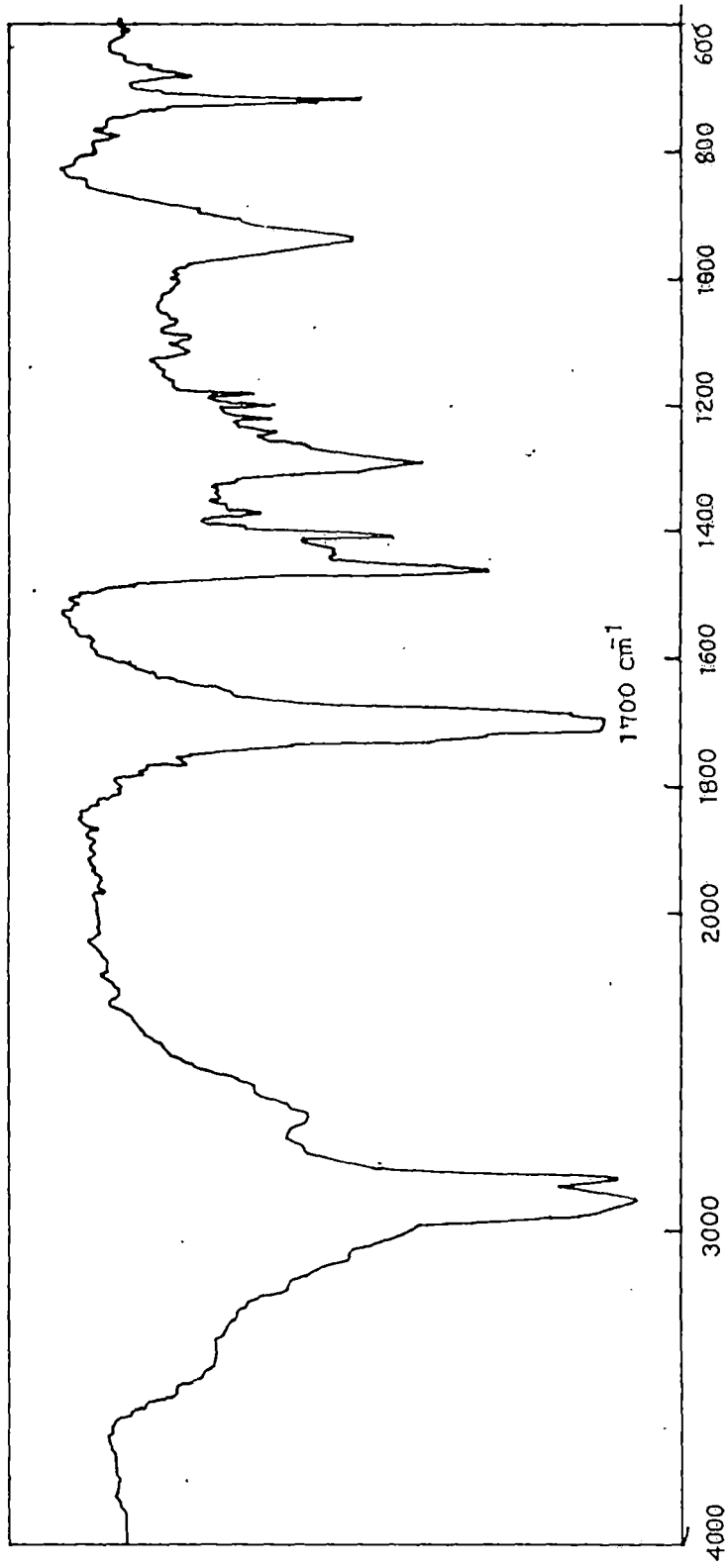


Fig.23. IR spectrum of tetracosanoic acid

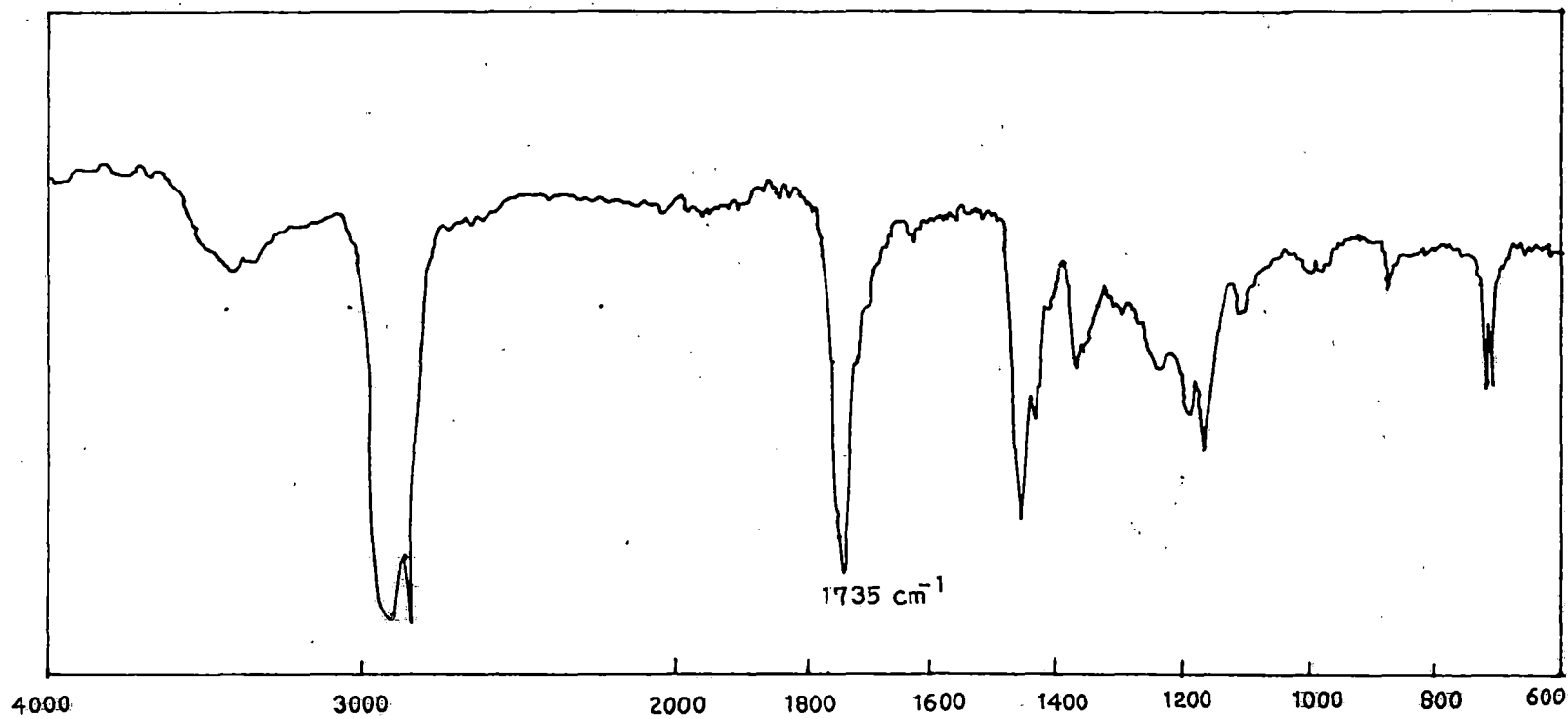


Fig 24. IR spectrum of methyltetracosanoate

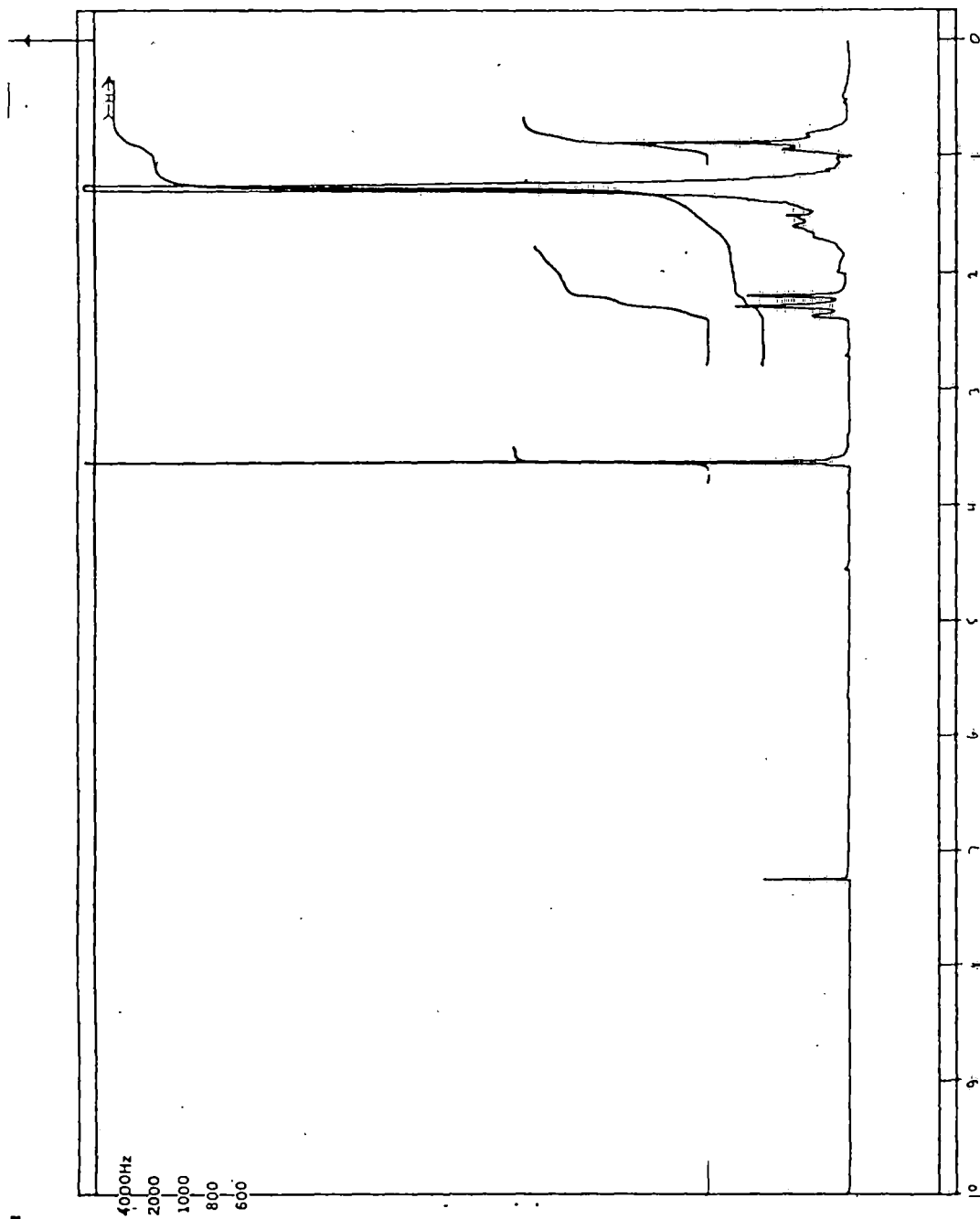


Fig 25. ^1H NMR spectrum of methyltetracosanate at 80 MHz

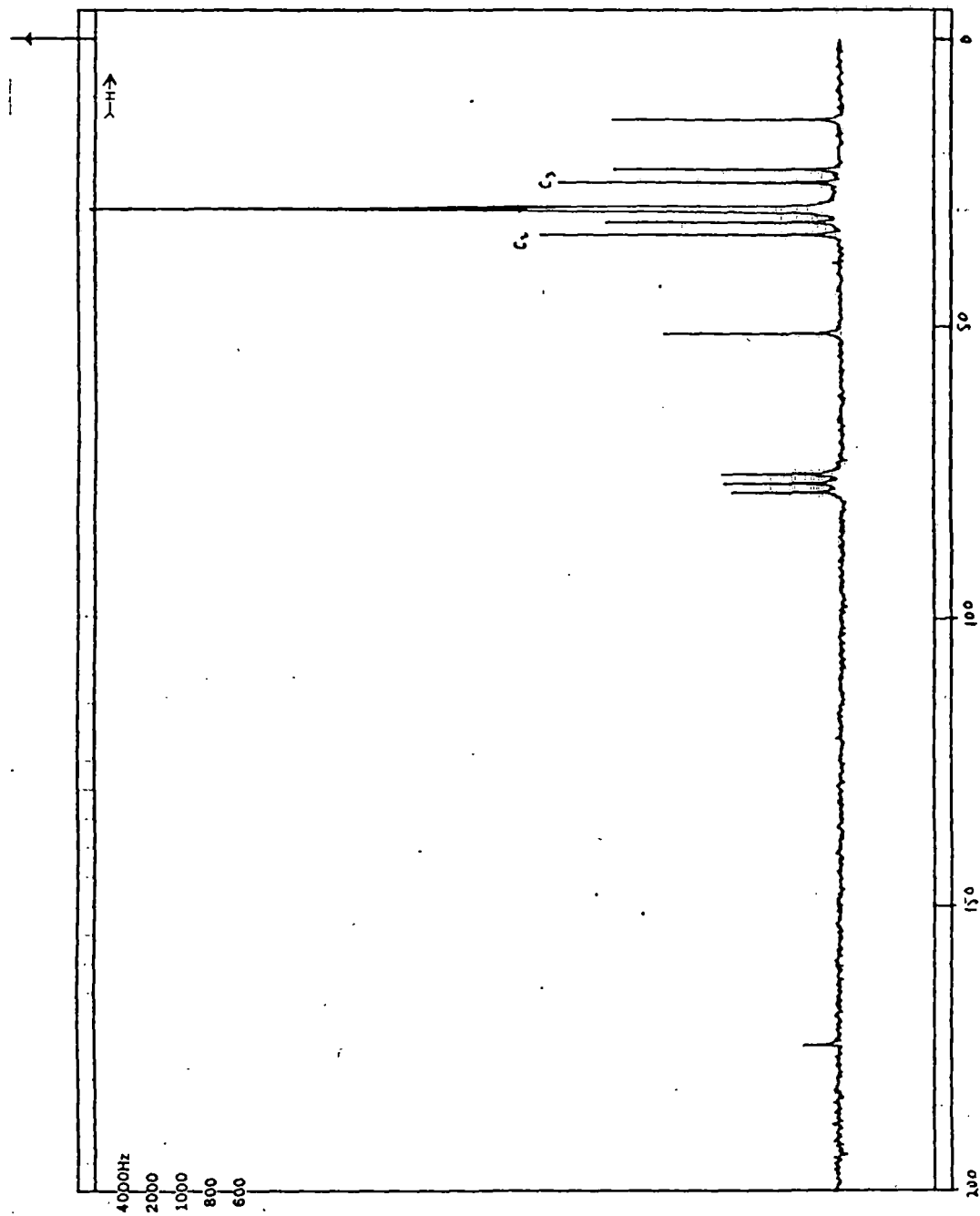


Fig 26. ^{13}C NMR spectrum of methyltracesanate at 20 MHz

Examination of fraction 2 (Table 6) : isolation and identification of benzoic acid

Fraction 2 was crystallized from hot water into crystalline solid, mp 121^o and was identified as benzoic acid by direct comparison with an authentic sample (mixed mp, co-TLC and co-IR).