

halide. The reaction has been extended to the 4-alkylnaphthols and the results are reported here.

The reaction of 4-methyl-1-naphthol² with carbon tetrachloride in the presence of anhydrous aluminium chloride, using molar proportions of the components, afforded the expected 4-methyl-4-trichloromethyl-1-(4H)-naphthalenone (I) isolated as a pale yellow liquid (b.p. 190/10 mm oil bath temperature) in about 5% yield. It gave the expected elemental analysis and its i.r. spectrum showed a carbonyl band at 1680 cm^{-1} . Its 2, 4-DNP and oxime were crystalline solids, m.ps. 224–25° and 140–41° respectively. The naphthalenone failed to undergo the rearrangements with PCl_5 or with PPA occurring with the corresponding 2, 5-cyclohexadienone derivatives¹.

4-Ethyl-1-naphthol³ (II) was prepared by the Clemmensen reduction of 4-acetyl-1-naphthol. During the reduction, besides II, small amounts of a yellow oil (yield 2%) was also isolated by chromatographic separation (benzene fraction). It was distilled at 200°/10 mm and TLC showed it to be a single substance (III). The elemental analysis and mass spectrum (M^+ 174, other peaks at m/e 145, 131, 117 and 91) indicated its molecular composition as $\text{C}_{12}\text{H}_{14}\text{O}$. The i.r. spectrum did not exhibit any OH band, but a distinct band was obtained at 1680 cm^{-1} showing the presence of a carbonyl group and this was confirmed by the formation of a crystalline 2, 4-DNP, m.p. 187–88°. The n.m.r. of III showed signals at δ 1.0 (3H, t, al, CH_3), δ 1.3 ~ 2.9 (7H, m, 3- CH_2 and CH), δ 7.2 ~ 7.7 (3H, m, C_5H , C_6H and C_7H), δ 8.1 (1H, m, C_8H).

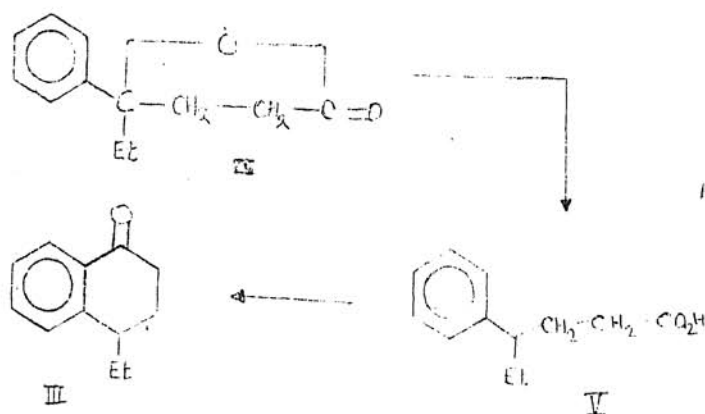
From the above data, the compound was assigned the structure as 4-ethyl-1, 2, 3, 4-tetrahydro-naphthalen-1-one (III). The latter compound has been described in literature⁴ as an oil but since it was not available for comparison purposes, it was synthesised by an unambiguous route as follows. Condensation of propiophenone and diethyl succinate in the presence of potassium under the conditions of Stobbe condensation afforded a half ester, which was refluxed with HBr in acetic acid to give the unknown γ -ethyl- γ -phenylbutyrolactone (IV). The latter was refluxed with HI and red phosphorus to yield the new acid (V) as an oil which on cyclisation with 80% sulphuric acid afforded the required tetralone as an oil (2, 4-DNP, m.p. 187–88°) identical with (III i.r., n.m.r., TLC).

The formation of the tetralone (III) during the Clemmensen reduction of 4-acetyl-1-naphthol is not entirely unexpected. It is of interest to note that Akram

REACTION OF 4-ALKYLNAPHTHOLS WITH CARBON TETRACHLORIDE

IN our earlier publications¹, we have reported the syntheses and the reactions of 4-alkyl-4-tribromoalkyl-2, 5-cyclohexadienones by the application of the Zincke-Suhl reaction involving the reaction of *p*-alkylphenols with carbon tetrachloride (or carbon tetrabromide) in the presence of the corresponding anhydrous aluminium

and Desai⁴ have reported the formation of 4-ethyl-ac-tetrahydro-1-naphthol during the Clam-mensen reduction of the above ketone, whereas the Russian authors³ have reported the isolation of 1-ethyl-1, 2, 3, 4-tetrahydronaphthalene during their reduction.



When the reactions of 4-ethyl-1-naphthol and 4-propyl-1-naphthol were attempted with carbon tetrachloride in the presence of anhydrous aluminium chloride under different experimental conditions, only a dark tarry mass was isolated in all cases, from which no pure product could be isolated.

All the compounds gave satisfactory elemental analysis.

The authors are grateful to the Ciba-Geigy Research Centre, Bombay, for the spectra and to Mrs. J. A. Patankar and Shri D. S. More for carrying out the microanalyses. The award of a CSIR Junior Research Fellowship to one of us (ARD) is gratefully acknowledged.

Department of Chemistry,
Institute of Science,
Bombay 400 032,
March 9, 1977.

J. R. MERCHANT.
R. G. JADHAV.
A. R. DESHPANDE.

1. Merchant, J. R. and Jadhav, R. G., *J. Ind. Chem. Soc.*, 1974, 51, 951 and reference therein.
2. Wenkert, E. and Stevens, T. E., *J. Am. Chem. Soc.*, 1956, 78, 5630.
3. Sergievskaya, S. I. and Morozovskaya, L. M., *J. Gen. Chem. (U.S.S.R.)*, 1944, 14, 1107; *Chem. Abstr.*, 1946, 40, 1786.
4. Akram, M. and Desai, R. D., *Proc. Ind. Acad. Sci.*, 1940, 11A, 154.