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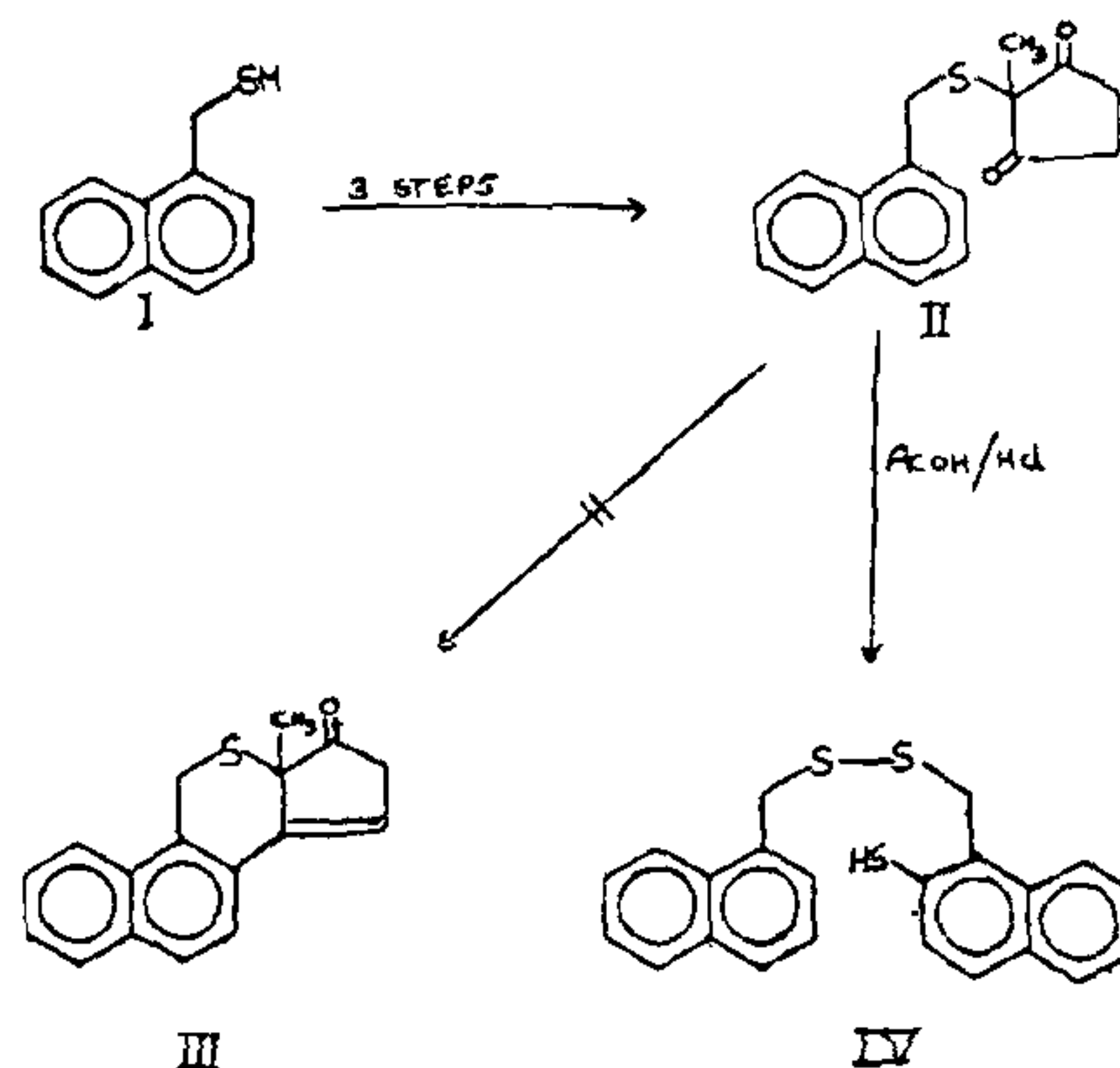
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**A NOVEL DISULPHIDE FORMATION
DURING THE CYCLODEHYDRATION OF
3-DEOXY-12-THIA-8,14-SECO-1,3,5(10),6,8-
ESTRAPENTAEN-14,17-DIONE**

RECENT reviews¹⁻⁴ on heterocyclic steroids indicate that there has been no report on the total synthesis and biological activity of 12-thiasteroids of aromatic and non-aromatic series. This prompted us to undertake the total synthesis of 3-deoxy-12-thia-1,3,5(10),6,8,14-estrapentaen-17-one(II) starting from 1-mercaptomethylnaphthalene(I)⁵.

Thus 3-deoxy-12-thia-8, 14-seco-1,3,5(10),6-8-estrapentaen-14,17-dione(II) was synthesized in three steps starting from 1-mercaptomethyl naphthalene as reported in our earlier communication⁶. Cyclodehydration of the secosteroid(II) employing the conventional catalysts such as methanolic hydrochloric acid, PTS in benzene, trifluoroacetic acid in chloroform, anhydrous SnCl₄ in benzene, etc., failed to furnish the anticipated steroid(III). However, attempted cyclodehydration of II with glacial acetic acid-hydrochloric acid (4 : 1) at 80-90°C for 8 hrs furnished, instead of the anticipated tetracyclic steroid, 1-naphthyl-methyl-2'-mercapto-1'-naphthylmethyl-disulphide(IV) as a

white crystalline solid, m.p. 134-35°, in 10% yield. The structure assigned to IV was evident from its spectral and analytical data; IR(CHCl₃): 2650 (W, SH), 1600, 1500 and 1400 cm⁻¹; ¹H NMR (CDCl₃);



1.5 (s, 1H, SH, proton), 3.9 (s, 2H, -CH₂-S-S-), 4.4 (s, 2H, -S-S-CH₂-), 7.8-8.2 (m, 13H, aromatic). The signal at 1.5 disappeared on shaking with D₂O and clearly indicated that it is an SH proton; ¹³C NMR (CDCl₃ δ): 44.04 (t, -CH₂-S-S), 44.32 (t, -S-S-CH₂-), (126.68-131.83) (aromatic carbons); Mass spectrum showed peaks at m/e 378 (M⁺, 2%), 232 (4%), 173 (6%), 171(5%), 142(10%), 141(100%); (Found C, 70.30, H, 5.13%. C₂₂H₁₈S₃ requires C, 69.84, H 4.76%).

The mechanism involving the formation of the product(IV) is under investigation.

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