

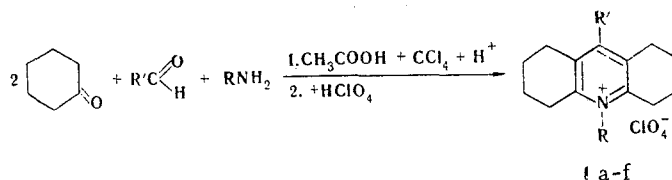
SYNTHESIS OF sym-OCTAHYDROACRIDINIUM SALTS

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The synthesis of sym-octahydroacridine from cyclohexanone, formaldehyde, and ammonium acetate at 180–200°C in 5% yield is known [1].

We have shown that N-substituted 9-aryl-sym-octahydroacridinium salts, which are isolated in the form of the perchlorates (Ia-f), are formed by refluxing (in xylene or benzene) cyclohexanone, an aromatic aldehyde, and a primary amine in a molar ratio of 2:1:1 with the addition of acetic acid, carbon tetrachloride (4 mole), and catalytic amounts of p-toluenesulfonic acid.



Ia R=R'=C₆H₅; b R=C₆H₄NO₂-p, R'=C₆H₅; c R=C₆H₄COOH-p, R'=C₆H₅; d R=C₆H₅, R'=C₆H₄OCH₃-p; e R=C₆H₅, R'=C₆H₄NO₂-p; f R=CH₂CH₂OH, R'=C₆H₅

The reaction apparently proceeds through the intermediate formation of N-substituted 9-aryldecahyde-acridines, inasmuch as condensation of cyclohexanone with benzaldehyde and aniline without acetic acid and CCl₄ gives 9,10-diphenyldecahydroacridine (detected chromatographically). Carbon tetrachloride acts as an oxidizing agent [2].

The yields in xylene are higher than in benzene, but the reaction is accompanied by resinification. Benzylidenecyclohexanone was obtained in appreciable quantities in the preparation of salt Ia; the yield of salt Ia is not increased when the mole fraction of cyclohexanone is increased. The IR spectra of salts I are in agreement with their structure; the IR spectrum of salt Ia is identical to that of a genuine sample [2]. The yields of salts I are negligible in the case of butyraldehyde, isobutyraldehyde, or chloral.

TABLE 1. N-Substituted 9-Aryl-sym-octahydroacridinium Perchlorates

Compound	mp, °C*	Empirical formula†	Yield, %
Ib	242–246	C ₂₅ H ₂₅ ClN ₂ O ₆	8
Ic	262–264 dec.	C ₂₆ H ₂₆ ClNO ₆	40
Id	250–251	C ₂₆ H ₂₈ ClNO ₅	18
Ie	153–154	C ₂₅ H ₂₅ ClN ₂ O ₆	17
If	222–223	C ₂₁ H ₂₆ ClNO ₅	20

*Alcohol is the solvent.

†All of the compounds have satisfactory elementary analyses (C, H, and N; only nitrogen was determined in the case of Ic).

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