PREPARATION OF 2-(N-MORPHOLINYL) INDENE AND ITS UNUSUAL STABILITY

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A method for preparing 2-(N-morpholinyl)-indene without using inert atmosphere and without requiring simultaneous removal of water has been described.

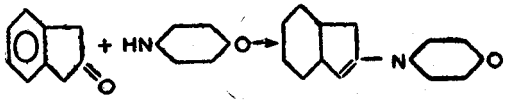
Enamines of 2-indanone were prepared earlier[1]. The method employed required the reactions to be carried under nitrogen atmosphere and simultaneous removal of water (azeotropically) formed during the course of reaction, which is the general method for preparing enamines of aldehydes and ketones[1,2]. In the present investigation while preparing enamine of 2-indanone (I) with morpholine, it was found that almost quantitative yields of 2-(N-morpholinyl)-indene (II) were obtained without using inert atmosphere and simultaneous removal of water was not required for the reaction to proceed.

The structure of 2-(N-morpholinyl)-indene (II) was established by elemental and spectral analysis (Fig. 1).

Elemental analysis of the compound (II) gave it the molecular formula $C_{13}H_{15}NO$ and NMR spectrum showed the presence of following signals (chemical shifts expressed in δ-ppm) :

6·8—7·3	ppm		Aromatic protons	Four
5·5	ppm	(singlet)	Olefinic protons	One
3·8	ppm	(triplet) (J9cps)	O-CH_2 protons	Four
3·5	ppm	(singlet)	-CH_2 protons	Two
3·05	ppm	(triplet) (J9cps)	N-CH_2 protons	Four

Enamines of ketones and aldehydes are described to be unstable and hydrolysable[1,2] by moisture. However, the enamine (II) was recovered unchanged even by boiling with water.



I II

Fig. 1—Structure of 2-(N-Morpholinyl)-indene by spectral analysis.

EXPERIMENTAL PROCEDURE

2-Indanone (*I*) :—2-Indanone (I) was prepared from indene by the method given by Rosen-et. al[3].

2-(N-Morpholinyl)-Indene (*II*) :—To 5·28 g (0·04 mol.) of freshly prepared and

dried 2-indanone, dissolved in purified dry benzene (50–70 ml), was added 6·96 g (0·08 mole.) freshly distilled morpholine. The mixture became warm and turbid. After keeping the mixture for about 30 minutes white crystalline solid (5·5 gms, m.p. 196°) settled which was filtered, washed with little benzene and washings were mixed with the filtrate. The filtrate was refluxed (1·5 hr), cooled and the crystalline solid (2·0 g) again obtained was filtered and washed with little benzene, (m.p. 196°). Chromatographic studies on thin layer of silica gel G using ethanol : pyridine : dioxane : water : : 50 : 20 : 25 : 5 : as moving phase revealed that both the solids were same having hrf value 69·16 (unstandardised). Crystallisation from hot benzene gave the pure compound II, 7·3 g, yield 91% m.p. 197° (reported[1] m.p. = 197°) (exposure of benzene solution to air turns it violet and avoided as far as possible).

Analysis : Calculated for $C_{13}H_{15}NO$; C, 77·61 ; H, 7·46 ; N, 6·96,

Found : C, 77·68 ; H, 7·32 ; N, 6·75.

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