Tetrahedron Letters No.32, pp. 2757-2762, 1965. Pergamon Press Ltd.
Printed in Great Britain.

CONFORMATIONAL ANALYSIS OF BENZOYLAMINOCYCLOHEXANE DERIVATIVES

Zen-ichi Yoshida

Department of Synthetic Chemistry, Kyoto University

Kyoto, Japan

Koji Nakagawa

Products Development Institute, Teijin Limited,

Iwakuni, Japan

(Received 17 June 1965)

In the present paper we report the experimental results of infrared absorption of benzoylaminocyclohexane (I), cis-1-benzoylamino-2-chlorocyclohexane (II) and trans-1-benzoylamino-2-chlorocyclohexane (III).

These compounds were synthesized according to the Schotten-Baumann reaction of coresponding cyclohexylamine derivatives and benzoylchloride. The infrared spectra of these compounds were measured on a Japan Spectroscopic Model DS-402G high resolution infrared spectrophotometer in crystalline state. The infrared spectra obtained are shown in Fig. 1, 2 and 3.

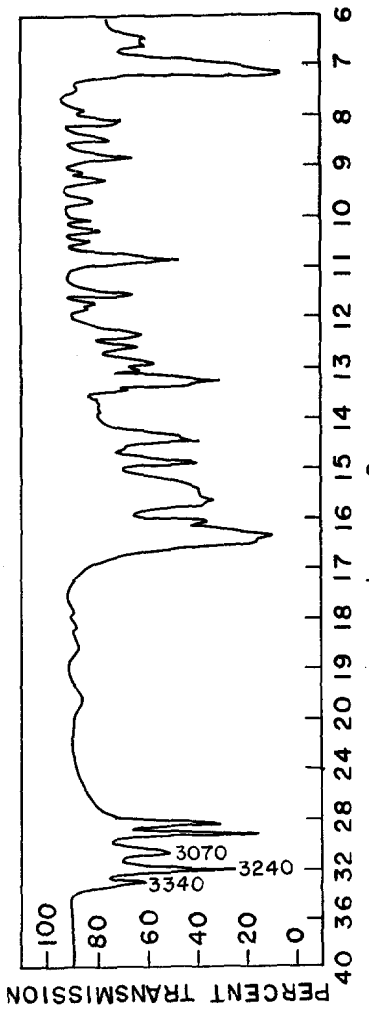

FIG. 1

Infrared absorpsion spectrum of benzoylaminocyclohexane (KBr tablet)

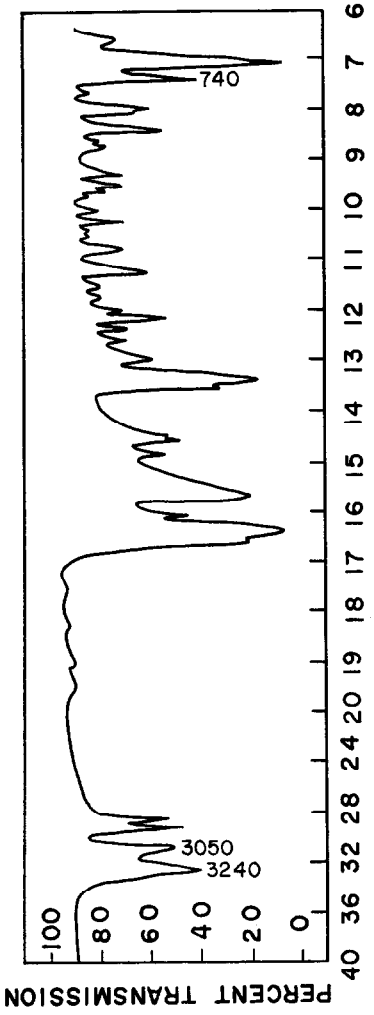

FIG. 2

Infrared absorption spectrum of trans-1-benzoylamino-2-chlorocyclohexane
(KBr tablet)

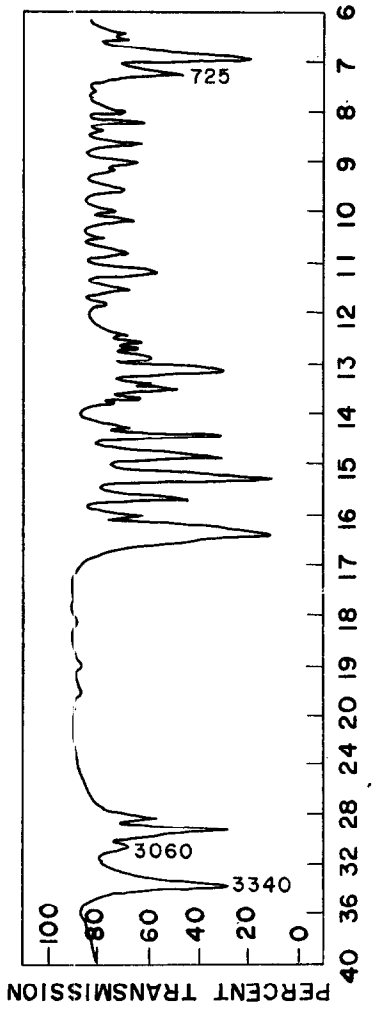

FIG. 3

Infrared absorption spectrum of cis-1-benzoylamino-2-chlorocyclohexane

From the comparison of 650 cm^{-1}-800 cm^{-1} region of these three spectra, it was concluded that 725 cm^{-1} band of (II) and 740 cm^{-1} band of (III) were C-Cl stretching vibration. These C-Cl stretching vibrations are assigned to equatorial C-Cl, for if they are due to axial C-Cl, they must be at lower frequency. For example in cyclohexyl chlorides the C-Cl absorptions are at 742 cm^{-1} (equatorial) and 688 cm^{-1} (axial).(1)* Accordingly conformations of (II) and (III) were determined as follows.

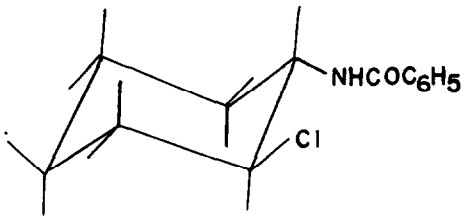

FIG. 4

Conformation of trans-1-benzoylamino-2-chlorocyclohexane

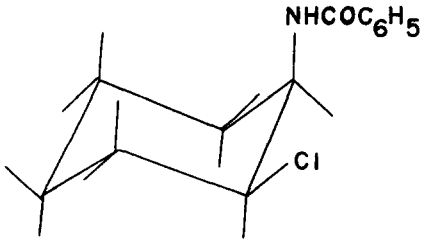

FIG. 5

Conformation of cis-1-benzoylamino-2-chlorocyclohexane

* Benzoylaminogroup is axial in (II) and equatorial in (III). Conformation of (I) could not be determined by this method.

NH association bands of (II) were observed at 3340 cm^{-1} and 3060 cm^{-1} and those of (III) at 3240 cm^{-1} and 3050 cm^{-1}. Therefore 3340 cm^{-1} band must be assigned to axial N-H and 3240 cm^{-1} band to equatorial NH. In the spectrum of (I) N-H stretching vibration was observed at 3340 cm^{-1}, 3240 cm^{-1} and 3070 cm^{-1}. The 3340 cm^{-1} band is assigned to axial NH and the 3240 cm^{-1} band to equatorial N-H reasonably. From these results conformation of (I) was determined as follows.

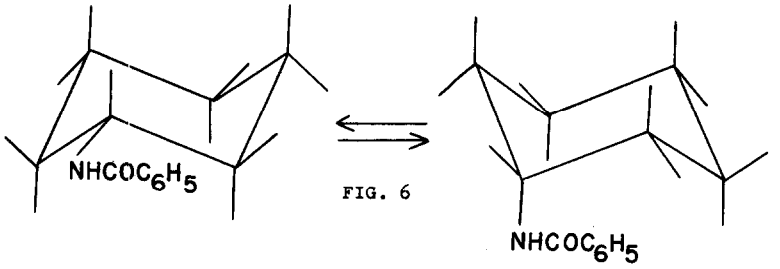

FIG. 6

Conformation of benzoylaminocyclohexane

Tuboi (2) has concluded that N-H association band of trans-amide is at 3370 cm^{-1}-3290 cm^{-1} and that of cis-amide is at 3240 cm^{-1}-3170 cm^{-1}.

Combination of his and our results leads to the conclusion that axial benzoylamino group is trans-amide and equatorial benzoylamino group is cis-amide.

References

1. Larnaudie, Compt. rend. 235, 154 (1952); 236, 909 (1953).
2. M. Tsuboi, Bull. Chem. Soc. Japan 22, 215 (1949).