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RESTRICTED ROTATIONAL ISOMERS II. MALEIMIDO DERIVATIVES OF THE cis AND trans ISOMERS OF 5-AMINO-2,4,6-TRIIODO-N,N,N',N'-TETRAMETHYLISOPHTHALAMIDES*

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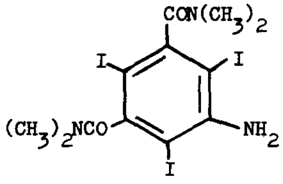
Our previous paper described the isolation of the cis and trans isomers of 5-amino-2,4,6-triiodo-N,N,N',N'-tetramethylisophthalamide (1) and the assignment of the cis and trans configurations by the preparation of the trans isomer in optically active form while the cis isomer was optically inert (1). We now wish to report that the nmr spectra of the maleimido derivatives of the cis and trans isophthalamides 1 also permit the assignment of the cis and trans structures.



1

When the cis and trans isomers of the isophthalamide 1 were heated on a steam bath with excess maleic anhydride for a short time and a small amount of sulfuric acid added to the molten mixture, cis- and trans-2,4,6-triiodo-5-maleimido-N,N,N',N'-tetramethylisophthalamide (2 and 3) were obtained, respectively. The formation of a cyclic imide from a 2,4,6-triiodoaniline was first reported by Holtermann who prepared N-(2,4,6-triiodophenyl) succinimide from 2,4,6-triiodoaniline (2). The maleimides 2 and 3 had mp's >360°, gave the correct analyses, had essentially identical uv spectra, had similar ir spectra, and showed single different spots with thin layer chromatography** at Rf 0.25 and 0.37,

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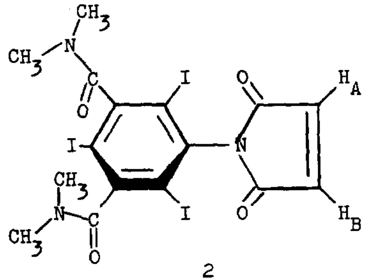
**Thin layer chromatography was done on pre-coated plates (Silica Gel F_{254}, E. Merck Ag.). An acetic acid: methanol: benzene (10:20:70) system was used.

respectively. The olefin regions of the nmr spectra were not identical when run in deuterated pyridine and are shown with the structural formulas.*



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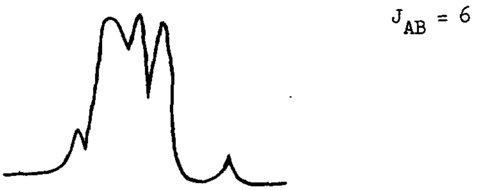

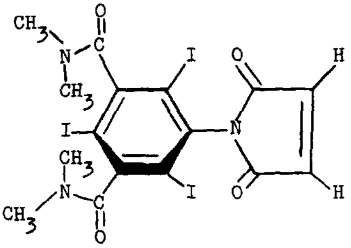

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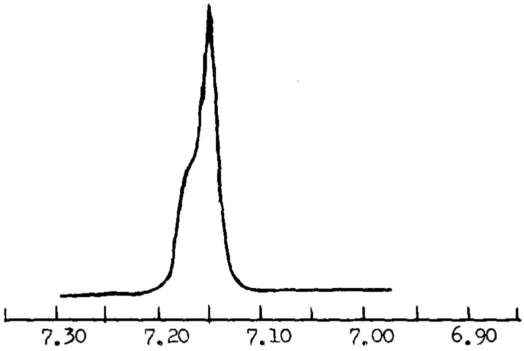

Examination of the structures of the maleimides 2 and 3 shows that the olefinic protons of the *cis* isomer 2 are in different magnetic environments and those of the *trans* isomer 3 are in identical magnetic environments. The nmr spectrum of the isomer 2 shows the nonequivalence of the olefinic protons and proves its *cis* configuration while the nmr spectrum of the isomer 3 shows a singlet for the olefinic protons and confirms its *trans* configuration.

Acknowledgement. The authors would like to thank Dr. R. K. Kullnig and his co-workers for the spectral data.

References.

(1) J. H. Ackerman, G. M. Laidlaw, and G. A. Snyder, *Tetrahedron Letters*, in press.
(2) Private communication from H. Holtermann, Oslo, Norway, 1964.

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*The nmr spectra were recorded on a Varian HA100 spectrometer and the olefin region was enlarged to examine this portion of the spectra. The broad signal at 7.17 is due to solvent impurity.