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**THE PHOTOTRANSPOSITION OF TRIMETHYLSILYL-N-METHYLPYRAZOLE**



**Miss Chanitsara Kocharit**

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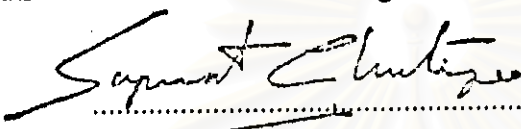
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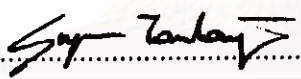
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ในการศึกษานี้ได้สังเคราะห์สารตั้งต้น 2 ชนิดคั้งนี้ 3-ไตรเมทิลไซลิท-1-เมทิลไพราโซล และ 5-ไตรเมทิลไซลิท-1-เมทิลไพราโซล และได้ศึกษาปฏิกิริยาโฟโตเคมีของสารทั้ง 2 ชนิดด้วย จากการคาดเดา โดยรูปแบบเพอมีวาทัน จะได้ว่าปฏิกิริยาโฟโตเคมีของ 5-ไตรเมทิลไซลิท-1-เมทิลไพราโซล ให้ผลิตภัณฑ์ 3 ชนิดคือ 5-ไตรเมทิลไซลิท-1-เมทิลอิมิดาโซล (P<sub>4</sub>) 2-ไตรเมทิลไซลิท-1-เมทิลอิมิดาโซล (P<sub>6</sub>) และ 4-ไตรเมทิลไซลิท-1-เมทิลอิมิดาโซล (P<sub>7</sub>) และปฏิกิริยาโฟโตเคมีของ 3-ไตรเมทิลไซลิท-1-เมทิลไพราโซล ให้ผลิตภัณฑ์ 2 ชนิดคือ 2-ไตรเมทิลไซลิท-1-เมทิลอิมิดาโซล (P<sub>4</sub> & P<sub>7</sub>) และ 4-ไตรเมทิลไซลิท-1-เมทิลอิมิดาโซล (P<sub>6</sub>) ทั้งนี้ยังได้สังเคราะห์ผลิตภัณฑ์ที่คาดว่าจะเกิดทั้งหมดนี้ด้วยเพื่อเป็นตัวเปรียบเทียบ แต่อย่างไรก็ตามการสังเคราะห์ 4-ไตรเมทิลไซลิท-1-เมทิลอิมิดาโซล ไม่ประสบผลสำเร็จ จากการศึกษาปฏิกิริยาโฟโตเคมีพบว่า 5-ไตรเมทิลไซลิท-1-เมทิลไพราโซล เกิดโฟโตทรานส์โพสิชันให้ผลิตภัณฑ์ 3 ชนิด และยังเกิดโฟโตคลิเวจให้ผลิตภัณฑ์อีก 1 ชนิด ผลิตภัณฑ์โฟโตทรานส์โพสิชันได้แก่ คือ ผลิตภัณฑ์หลัก 5-ไตรเมทิลไซลิท-1-เมทิลอิมิดาโซล (P<sub>4</sub>) 4-ไตรเมทิลไซลิท-1-เมทิลอิมิดาโซล (P<sub>7</sub>) และ 1-เมทิลอิมิดาโซล ซึ่งคาดว่าเปลี่ยนรูปมาจาก 2-ไตรเมทิลไซลิท-1-เมทิลอิมิดาโซล (P<sub>6</sub>) ส่วนผลิตภัณฑ์โฟโตคลิเวจคือ 3-(ไตรเมทิลไซลิท)-3-(เอ็นเมทิลอะมิโน)ไพโรพีนไนโตรล

3-ไตรเมทิลไซลิท-1-เมทิลไพราโซล เกิดโฟโตทรานส์โพสิชันให้ผลิตภัณฑ์ 2 ชนิด และยังเกิดโฟโตคลิเวจให้ผลิตภัณฑ์อีก 1 ชนิด ผลิตภัณฑ์โฟโตทรานส์โพสิชันได้แก่ 4-ไตรเมทิลไซลิท-1-เมทิลอิมิดาโซล (P<sub>6</sub>) และ 1-เมทิลอิมิดาโซล ซึ่งคาดว่าเปลี่ยนรูปมาจาก 2-ไตรเมทิลไซลิท-1-เมทิลอิมิดาโซล (P<sub>4</sub> & P<sub>7</sub>) ส่วนผลิตภัณฑ์โฟโตคลิเวจคือ 3-(เอ็นเมทิลอะมิโน)ไพโรพีนไนโตรล ซึ่งเป็นผลิตภัณฑ์หลักของปฏิกิริยานี้ นอกจากนี้แล้วยังได้สังเคราะห์ 4-ไตรเมทิลไซลิท-1-เมทิลไพราโซล ซึ่งเป็นสารตั้งต้นที่สำคัญอีกชนิดหนึ่ง แต่มีความบริสุทธิ์ไม่เพียงพอต่อการทำปฏิกิริยาโฟโตทรานส์โพสิชัน

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ลายมือชื่ออาจารย์ที่ปรึกษา .....

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In this study, photoreactants, 3- and 5-(trimethylsilyl)-1-methylpyrazoles were synthesized and their photoreactions were investigated. Predicted by permutation pattern, 5-(trimethylsilyl)-1-methylimidazole ( $P_4$ ), 2-(trimethylsilyl)-1-methylimidazole ( $P_6$ ), and 4-(trimethylsilyl)-1-methylimidazole ( $P_7$ ), the three predicted photoproducts of 5-(trimethylsilyl)-1-methylpyrazole, and 2-(trimethylsilyl)-1-methylimidazole ( $P_4$  &  $P_7$ ) and 4-(trimethylsilyl)-1-methylimidazole ( $P_6$ ), the two predicted photoproducts of 3-(trimethylsilyl)-1-methylpyrazole were synthesized as the authentic compounds for comparison. However, attempt to synthesize 4-(trimethylsilyl)-1-methylimidazole was unsuccessful. It was found that 5-(trimethylsilyl)-1-methylpyrazole underwent phototransposition to three photoproducts as well as photocleavage to the other product. The phototransposition products were 5-(trimethylsilyl)-1-methylimidazole as a major product of  $P_4$  process, 4-(trimethylsilyl)-1-methylimidazole as a predicted product of  $P_7$  process, and 1-methylimidazole. The last product was assumed to be the secondary product arising from 2-(trimethylsilyl)-1-methylimidazole which was a  $P_6$  photoproduct. The photocleavage product obtained as well, was identified as 3-(trimethylsilyl)-3-(*N*-methylamino)propenenitrile.

3-(Trimethylsilyl)-1-methylpyrazole underwent phototransposition to two photoproducts as well as photocleavage to the other product. The phototransposition products were 4-(trimethylsilyl)-1-methylimidazole as a predicted product of  $P_6$  process and 1-methylimidazole which was assumed to be the secondary product arising from 2-(trimethylsilyl)-1-methylimidazole as a product of  $P_4$  &  $P_7$  processes. The reaction also gave a photocleavage product which was identified as 3-(*N*-methylamino)propenenitrile. Furthermore, 4-(trimethylsilyl)-1-methylpyrazole, the other isomer of the photoreactants of this series, was also synthesized. Unfortunately its low purity was not suitable to carry out the phototransposition reaction.

ภาควิชา.....เคมี

สาขาวิชา.....เคมี

ลายมือชื่อนิสิต.....

ลายมือชื่ออาจารย์ที่ปรึกษา..... *Sup Tantay*

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.....

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สถาบันวิทยบริการ  
จุฬาลงกรณ์มหาวิทยาลัย

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## LIST OF ABBREVIATIONS

TMS	Tetramethylsilane
ppm	part per million
$\delta$	chemical shift
s	singlet (NMR)
d	doublet (NMR)
t	triplet (NMR)
q	quartet (NMR)
$\text{cm}^{-1}$	unit of wave number
m/z	mass to charge ratio
$\lambda_{\text{max}}$	the wavelength at maximum absorption
%	percent
bp.	boiling point
mp.	melting point
HMPT	hexamethylphosphoric triamide
THF	tetrahydrofuran

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จุฬาลงกรณ์มหาวิทยาลัย