# Synthesis of Alumatrane Complexes Directly from Al(OH)<sub>3</sub> and Triisopropanolamine

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#### **ABSTRACT**

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**HYDROXIDE** 

YUKOLTORN OPORNSAWAD: SYNTHESIS OF
ALUMATRANE COMPLEXES DIRECTLY FROM Al(OH)<sub>3</sub>
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Preparations of alumatrane complexes generally are high cost because of multistep synthesis and expensive starting materials. Recently, a new one step method was developed for synthesizing alumatrane directly from aluminum hydroxide [Al(OH)<sub>3</sub>] and triisopropanolamine (TIS) both of which are inexpensive and readily available. When 45.5 mmol of Al(OH)<sub>3</sub> are reacted with 70 mmol of TIS at 200°C, the reaction is complete in 3 h. The product can be purified by precipitation. Triethylenetetramine (TETA), a stronger base than TIS, was found to accelerate the dissolution rate of Al(OH)<sub>3</sub>.

The kinetics of TIS-Al formation were studied and TIS-Al was fully characterized using DSC, TGA, FAB<sup>+</sup>-MS, NMR (<sup>1</sup>H-, <sup>13</sup>C-, <sup>27</sup>Al-), and FTIR. The integral method was used to study the dissolution kinetics as a function of different conditions. The activation energy of reaction was 24±2 kJ mol<sup>-1</sup>.

#### บทคัดย่อ

ยุคลธร โอพรสวัสดิ์ : การสังเคราะห์สารประกอบอลูมาเทรนโดยตรงจากอลูมีนั้ม ใชครอกไซด์ และไตรไอโซโพรพาโนลามีน [Synthesis of Alumatrane Complexes Directly from Al(OH), and Triisopropanolamine], อาจารย์ที่ปรึกษา : Assoc. Prof. Richard M. Laine และ ผศ. คร. สุจิตรา ธำรงวราภรณ์, 42 หน้า, ISBN 974-633-595-2

โดยทั่วไปแล้ว การเตรียมสารประกอบอลูมาเทรนมักต้องเสียค่าใช้จ่ายที่สูง ทั้งนี้เพราะเป็น การสังเคราะห์ที่ค่อนข้างยุ่งยากและสารตั้งค้นที่ใช้มีราคาแพง เมื่อเร็ว ๆ นี้ ได้มีการพัฒนาวิธีการ สังเคราะห์สารประกอบอลูมาเทรนด้วยวิธีใหม่ที่สะควกและประหยัดจากอลูมินัมไฮครอกไซค์ และ ไตรไอโซโพรพาโนลามีน ซึ่งสารตั้งค้นทั้งสองมีราคาไม่สูง และหาได้ง่าย โดยนำอลูมินัมไฮครอกไซค์ 45.5 มิลลิโมล มาทำปฏิกิริยากับไตรไอโซโพรพาโนลามีน 70 มิลลิโมล ที่อุณหภูมิ 200 องศาเซลเซียส ปฏิกิริยาเสร็จสมบูรณ์ภายในเวลา 3 ชั่วโมง นำผลิตภัณฑ์ที่ได้ไปทำให้บริสุทธิ์โดยการตกตะกอน นอกจากนี้ยังได้พบว่าใตรเอทธิลีนเตทตระมีนซึ่งเป็นเบสที่แรงกว่าใตรไอโซโพรพาโนลามีนเป็นตัวเร่ง อัตราการละลายของอลูมินัมไฮครอกไซค์อีกด้วย

ได้มีการศึกษาจลนศาสตร์ของการเกิดสารประกอบอลูมาเทรน และการตรวจสอบโครงสร้าง พร้อมทั้งศึกษาสมบัติของผลิตภัณฑ์โดยใช้ DSC, TGA, FAB -MS, NMR (H-, 13C-, 27Al-), และ FTIR ได้นำวิธีอินทีกรัลมาใช้ในการศึกษาจลนศาตร์ของการละลายในตัวแปรต่าง ๆ พลังงาน กระตุ้น ของปฏิกิริยามีค่าเท่ากับ 24±2 กิโลจูล ต่อโมล

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