Spectroscopy, chromatography and microscopic image of 3-(4-methoxyphenyl)-1-phenylprop-2-en-1-one (MNYAD_1539) crystals

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ABSTRACT

3-(4-Methoxyphenyl)-1-phenylprop-2-en-1-one (MNYAD_1539) was synthesized by an Aldol condensation method. MNYAD_1539 was synthesized from two commercially available materials of p-Methoxybenzaldehyde and Acetophenone. The physical and chemical properties of MNYAD_1539 was investigated using ATR-FTIR, GCMS and a microscope. The FTIR spectrum of the crude MNYAD_1539 showed the presence of impurities, compared with the crystalline sample. The chromatogram showed that MNYAD_1539 with high purity was produced after recrystallisation. The current study also found high quality of the transparent needle-like crystal after re-crystallisation.

INTRODUCTION

The first synthesis of 3-(4-methoxyphenyl)-1-phenylprop-2-en-1-one (MNYAD_1539) was made in 1913. After that, many new synthesis methods for the preparation of MNYAD_1539 were explored such as with oxidation, dehydration and hydrolysis reaction. However, the most popular method is condensation reaction because of the high yield of the obtained product.1,2

The physical data of MNYAD_1539 in solid state including melting point and crystal colours has been determined. Also, the molecular vibrations of MNYAD_1539 was investigated by FTIR in KBr matrix.3

To the best of our knowledge, there is no microscopic images and ATR-FTIR spectrum in it format has been reported. Hence, the current work describes the synthesis and characterization of MNYAD_1539. The crystals are obtained after the re-crystallization of the crude.

RESULTS AND DISCUSSION

Base catalyzed Aldol condensation of MNYAD_1539:

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\begin{align*}
\text{NaOH} & \quad \text{+MNYAD_1539} \quad \text{Recrystallization with EtOH} \quad \text{+ H}_2\text{O} \\
\text{Re} & \quad \text{condensation reaction} \\
\text{GCMS and ATR-FTIR Charaterization of MNYAD_1539 crystals :} \\
\text{ATR-FTIR SPECTRA} & \quad \text{GC CHROMATOGRAM} \\
\text{Figure 2. Solid state IR spectra of MNYAD_1539 before crystallization (top) and after crystallization (bottom). The presence of additional peak at 1681 cm}^{-1} \text{ in the crude product might attributed to the reactants. Also, broad peak at 3538 cm}^{-1} \text{ indicate the v(O-H) very likely from water. However, they are absence after crystallization.} \\
\text{Expansion of IR region above 1800 cm}^{-1} \\
\text{Figure 3. An intense peak was observed in the GC-MS chromatogram of the MNYAD_1539 solution. The solution also containing a very low level of another compound. These suggests a mixture of unequal composition was obtained after the chemical reaction.} \\
\text{Figure 4. The first and second peak showed very similar mass fragmentation with molecular ion of 238 which indicates a very similar compound. MNYAD_1539 compound containing C_{16}H_{16}O_3 (CAS : 955-33-1) with the help of NIST library.} \\
\text{Figure 5. Images under microscope (40x) showing transparent needle-like morphology of the MNYAD_1539 after recrystallization from ethanol. The melting point is 86 °C and this yellow sample has rubber melting-like smell. These images are first reported for this compound.}
\end{align*}
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METHODOLOGY

SYNTHESIS

CRISTALLIZATION

RECRYSTALLIZATION

STRUCTURAL STUDY

GC-MS

MICROSCOPY

ATR-FTIR

CONCLUSION

• 3-(4-methoxyphenyl)-1-phenylprop-2-en-1-one was successfully synthesised and characterised by vibrational spectroscopy, gas chromatography mass spectrometry and microscopic analysis.

• Its needle-like crystals were successfully grown after recrystallization from ethanol.

• The crystal of the compound was successfully observed under microscope showing transparent needle-like and it has rubber melting-like smell. The melting point of the crystal is 86 °C.

• An very intense peak was observed in the chromatogram and it also contains a very low level of similar compound.

REFERENCES


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ACKNOWLEDGEMENT

This work was supported by the Department of Chemistry, Faculty of Science at UPM as part of the undergraduate laboratory practices.