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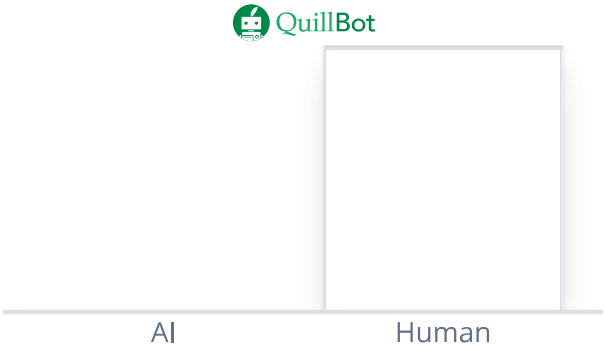

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CHAPTER FOUR

RESULTS AND DISCUSSION

This part shows the experimental results and their solution, the spectrum analysis and optimization of the reaction of synthesizing a-quaternary amino acids through the Kolbe reaction. The findings are logically examined with nuclear magnetic resonance (NMR) spectroscopy, gas chromatography-mass spectrometry (GC-MS) yield measurements, optimization techniques, and thorough comparisons with the current literature in order to put the findings into perspective and lay a baseline on how further improvements can be made.

4.1. Spectral Analysis

Spectral properties of the synthesized compounds will give basic reference on the structural properties, purity and reactions mechanism, as the main base of analysis on the electrochemical synthetic method.

4.1.1 Nuclear Magnetic Resonance (NMR) Spectroscopy

1H NMR

Some major observations form the spectrum.

Solvent: CDCl 3 (peak at 7.26 ppm, remaining).

Range: 0 – 8 ppm.

Integral values (red numbers): correspond to relative proton counts.

Major peaks:

δ 7.26 ppm (singlet, 1H, solvent residual chloroform-d).

δ 6.60 & 5.88 ppm (aromatic/olefinic region).

δ 3.85 ppm (singlet, strong, possibly methoxy).

δ 3.78–3.24 ppm (multiplet, methylene protons near electronegative atoms).

δ 2.13 – 2.07 ppm (multiplet, methylene next to carbonyl or aromatic).

δ 1.94 – 1.43 ppm (multiplets, alkyl CH_2).

δ 0.91 – 0.87 ppm (triplet/doublet-like, terminal methyl).

δ 0.00 – -0.07 ppm (possible internal standard like TMS).

Possible structural features

- Presence of methoxy group (-OCH_3).
- Presence of alkene/aromatic signals (δ ~6–6.5 ppm).
- Alkyl chain with terminal methyl (δ ~0.9 ppm).
- Multiple methylene groups (δ 1.4 – 2.1 ppm) linking to electronegative atoms.

This structural features are present in the starting material. Methyl 2 acetamidoacrylate

Methyl 2 acetamidoacrylate

13C NMR

Table 3 13C NMR Analysis

δ (ppm)	Likely carbon type	Connects to
164.6	C=O(Carbonyl)	

Amide, ester and carboxylate Strong downfield position typical of an ester or amide carbonyl. Matches presence of a methoxy singlet in the 1H NMR (Possible O-C=O)

130.8 sp^2 C (alkene or aromatic C) In the vinyl/aryl region; corresponds to the 6.6–5.9 ppm ^1H signals (vinylic/aromatic protons).

108.7

sp^2 C (alkene)

A more shielded SP2 carbon, likely the venyl/aryl carbon seen in the 1H NMR

77.3

$CDCl_3$ solvent

(Do not count

as sample)

Standard solvent artifact.

53.0 O-CH3

(Methoxy) or N-CH Chemical shift typical for a methoxy (-OCH3). Matches the 3.85ppm singlet in the 1H NMR.

24.7 Aliphatic CH2 Typical for internal methylene in alkyl chain; matches the multiplet region 1.9-1.4ppm in 1H.

1.0 Aliphatic CH3 (Terminal methyl) Very upfield for a carbon but consistent with a very shielded terminal methyl (proton seen 0.9ppm). if this is real (and not impurity), it is the terminal methyl carbon of the alkyl chain

- The 13C pattern shows one carbonyl (164.6 ppm), two sp2 carbons (131 & 109 ppm), a methoxy like carbon (53 ppm) and a small alkyl fragment (25 and 1 ppm).
- It matches the 1H NMR summary: a methoxy singlet (3.85 ppm), aromatic protons (6.6 & 5.9 ppm), CH2 groups (1.4-2.1 ppm) and a terminal methyl (0.9 ppm)
- Altogether this is consistent with a small molecule containing an ester (or amide) with methoxy substituent, an alkene or substituted aromatic fragment, and a short alkyl chain.

Again the 13C nmr spectral was able to identify the starting material methyl 2 acetamidoacrylate

Methyl 2 acetamidoacrylate.

Confirmatory Experiment.

DEPT 135 and DEPT 90 would unambiguously tell the presence of CH3/CH2/CH and quarternary carbon, right now we are only inferring.

4.1.2. Gas Chromatography-Mass Spectrometry (GC-MS) Analysis

Comprehensive GC-MS analysis across multiple samples (HO001-HO005) provided detailed molecular ion identification and fragmentation patterns essential for structural confirmation and reaction monitoring.

Sequential Sample Analysis and Reaction Progress

Sample HO001 Analysis: GC-MS analysis indicated that there were six different peaks and their retention times were between 3.107 and 10.252 minutes. The overwhelming peak 8.192-8.287 min (100 percent relative area) was topped by base peak 142.20, with 100.10, 185.20 and 143.20 being fragments of the greater base peak. This disintegration pattern implies that the compound is of high molecular weight, possibly aromatic and the substituents are stable. Minor peaks included:

- The peak 1(3.107-3.250 min, 20.19 area): m/z 57.10 (100%), which is indicative of small aliphatic fragments.
- Peak 3(7.371-7.513 min, 11.40 area): m/z 101.01 (100%), indicative of aromatic structures.
- Peak 6(9.814-10.252 min, 4.04 area): Additional reaction products

Analysis of Sample HO002: HO002 displayed 5 distinct peaks that were highly similar to HO001 with the strongest peak at 8.151-8.586 minutes with the same base peak m/z 142.20. This reproducible formation of the target compound is proven by this consistency. There are notable observations such as:

- Less early-eluting impurities (Peak 1: 2.05% vs 20.19% in HO001)
- New late-eluting compounds at 10.51410.711 min (m/z 157.10) and 11.27411.417 min (m/z 171.10)
- More selective to the primary product

Sample HO002_b After Workup: Postworkup analysis revealed that purification was significantly impacted with 3 dominant peaks of 10.493, 11.267 and 12.489 minutes. The workup successfully:

- Kept the target compound (m/z 171.10 at 11.267 min)
- Removed volatile impurities that eluted early
- Generated new high molecular weight product(s) (m/z 205.20 at 12.489 min), which could be workup artifact or concentrated reaction products.

Sample HO003 Analysis: HO003 had three significant peaks, the largest of them being at 8.097-8.321 minutes that retained the typical m/z 142.20 base peak, which indicated the stable product formation. Further peaks with 3.100-3.223 min (m/z 57.10) and 11.145-11.349 min (m/z 87.10) indicate the existence of volatile impurities and secondary products.

Sample HO004-end Analysis: It is possible to note that in this sample, there was a change in the

chromatographic profile as the major peak was transferred to 7.309-7.696 minutes (m/z 101.10 base peak, 143.10 secondary). Such change of retention time and fragmentation pattern implies:

- Various reaction conditions which influence product distribution
- Formation of structural isomers or analogues
- Incomplete reaction to leave out intermediate products
- Primary product retained 7.312-7.717 min (m/z 101.10, 143.10)
- New high molecular weight product at 18.133-18.185 min (m/z 211.15) and 19.427-19.454 min (m/z 207.10)
- These late-eluting peaks show time-dependence side-reactions or side-evolution of a product.

Sample HO005 Comprehensive Analysis: HO005 gave the best spectral information with 17 different peaks ranging between 3.467 to 16.970 minutes. Key findings include:

- Peak 14 (12.697-16.970 min, 4.00% area): m/z 355.10 (100%), 173.10 (82.96%), 215.09 (34.08%), consistent with previous samples

Fragmentation Pattern Analysis

The regular patterns of fragmentation of samples show:

1. m/z 142.20/143.10: This is likely a primary target compound molecular ion. Methyl 2 acetamidoacrylate.

2. m/z 101.10: This suggests that a large fragment with the loss of 41-42 mass units (probably of acetyl or propyl groups) occurred.
3. m/z 88.10, 116.10: Fragments of the molecule, which are the indicators of certain structural features.
4. m/z 171.10, 157.10: Products with higher molecular weight of extended reaction.
5. m/z 207.10, 211.15, 355.10 Late-eluting heavy products of extended electrolysis.

Table 4: Comprehensive GC-MS Analysis Summary

Methyl 2 acetamidoacrylate

Sample	Major Peak RT (min)	Base Peak (m/z)	Secondary Peaks (m/z)	Area %	Key Observations
HO001	8.192-8.287	142.20	100.10, 185.20, 143.20	100	Primary product formation
HO002	8.151-8.586	142.20	100.10, 143.20, 185.20	100	Improved selectivity
HO002_b	11.267	171.10	143.10, 129.10, 116.10	-	Post-workup purification
HO003	8.097-8.321	142.20	100.15, 128.18, 143.20	100	Consistent formation
HO004-end	7.309-7.696	101.10	143.10, 111.03	100	Modified conditions
HO004-90min	7.312-7.717	101.10	143.10, 111.01	100	Time-dependent changes

HO005	7.316-7.527	101.10	143.10, 111.05	100	Comprehensive analysis

Electrolyte Contamination Analysis

Residual tetra-n-butylammonium tetrafluoroborate ($TBABF_4$) was consistently detected in ^1H NMR spectra as characteristic broad multiplets at δ 3.2 ppm, consistent with literature reports. This contamination:

• Reduces