*Supporting Information*

# Iminoxyl radicals vs. *tert*-butylperoxyl radical in competitive oxidative C-O coupling with β-dicarbonyl compounds. Oxime ether formation prevails over Kharasch peroxidation

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## 1. General

$^{1}H$ and $^{13}C$ NMR spectra were recorded on Bruker AVANCE II 300 spectrometer (300.13 MHz and 75.48 MHz, respectively) in $CDCl_3$. Chemical shifts were reported in parts per million (ppm), and the residual solvent peak was used as an internal reference: $^{1}H$ ($CDCl_3$ δ=7.26 ppm), $^{13}C$ ($CDCl_3$ δ=77.16 ppm). Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet). Coupling constants were reported in Hertz (Hz).

High resolution mass spectra (HR-MS) were measured on a Bruker maXis instrument using electrospray ionization (ESI). The measurements were performed in a positive ion mode (interface capillary voltage – 4500 V); mass range from *m*/*z* 50 to *m*/*z* 3000 Da; external calibration with Electrospray Calibrant Solution (Fluka). A syringe injection was used for all acetonitrile solutions (flow rate 3 μL/min). Nitrogen was applied as a dry gas; interface temperature was set at 180 °C.

FT-IR spectra were recorded on a Bruker ALPHA FT-IR spectrometer.

Cyclic voltammograms were recorded and controlled potential electrolysis were implemented with an IPC-Pro potentiostat (Econix) (the accuracy of the scan rate is 1.0 %; the accuracy of the potential setting is 0.25 mV). Experiments were performed in a 10-mL five-neck glass conical-shaped electrochemical cell with a water jacket for thermal control. The glassy carbon disk electrode (*d* = 1.7 mm) served as the working electrode; a platinum wire as the auxiliary electrode. A silver chloride electrode (Ag/AgCl) served as the reference electrode, which was linked to the solution under study by a bridge with a porous ceramic diaphragm filled with the supporting electrolyte. The potentials were corrected for the ferrocene oxidation potentials under the same conditions (0.49 V *vs.* Ag/AgCl). The solutions were kept under thermally controlled conditions at 25±0.5 °C and deaerated by bubbling argon. Electrochemical experiments were performed under an argon atmosphere.

The EPR spectra were recorded on a Bruker ER-200D spectrometer in a X-band range (ν~9600 MHz). The magnetic field strength was measured with an accuracy of ± 0.02 G using an ER 035M magnetometer built in the EPR spectrometer. The microwave frequency in the cavity was determined using a ChZ-46 frequency meter with an accuracy of ± 0.1 MHz.

Column chromatography was performed using silica gel (0.060-0.200 mm, 60 Å, Acros).

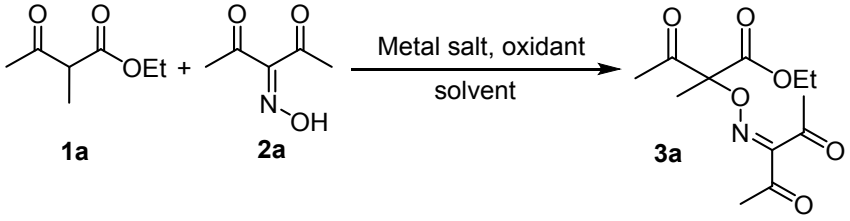
$CH_2Cl_2$ was distilled prior to use. EtOAc and MeCN were distilled over $P_2O_5$. Glacial acetic acid was used as is from commercial sources.

$Cu(BF_4)_2$ hydrate (anhydrous basis purity ca. 68%, corresponds to approximate formula $Cu(BF_4)_2{\cdot}6H_2O$), $Cu(ClO_4)_2{\cdot}6H_2O$, $CuCl_2$, CuI, CuCl, $CuSO_4{\cdot}5H_2O$, $Cu(OAc)_2$, $Cu(OTf)_2$, $(NH_4)_2CuCl_4{\cdot}2H_2O$, $Mn(OAc)_3{\cdot}2H_2O$ 95%, $Mn(ClO_4)_3{\cdot}6H_2O$, $Fe(ClO_4)_3$ hydrate (anhydrous

basis purity ca. 65%, corresponds to approximate formula $Fe(ClO_4)_3 \cdot 11H_2O$), $Ni(OAc)_2 \cdot 4H_2O$, $AgNO_3$, tetrabutylammonium iodide, 2,2'-bipyridine, *tert*-butylhydroperoxide (70% aqueous solution), *tert*-butylhydroperoxide (5.0-6.0 M solution in decane), $H_2O_2$ (34% aqueous solution), di-*tert*-butylperoxide, (diacetoxyiodo)benzene, $(NH_4)_2S_2O_8$, $K_2S_2O_8$, Oxone ($KHSO_5 \cdot 0.5KHSO_4 \cdot 0.5K_2SO_4$), ethyl 2-methylacetoacetate **1a**, 2-acetylbutyrolactone **1b** and diethyl acetylsuccinate **1d** were commercial reagents (Acros, Sigma-Aldrich, Alfa Aesar). All the other β-dicarbonyl compounds[1-5] and all the oximes[6-10] were synthesized according to the literature.

## 2. Optimization of oxidative C-O coupling of β-keto ester 1a with oxime 2a (full Table 1 and experimental details)

**Table S1.**




**1a** **2a** **3a**

| Entry | Metal salt (mole per mole of **1a**) | Oxidant (mole per mole of **1a**) | Solvent | Yield of **3a** (%) |
|---|---|---|---|---|
| 1 | $Cu(BF_4)_2 \cdot 6H_2O$ (0.1) | $O_2$ | MeCN | 14 |
| 2 | $Cu(BF_4)_2 \cdot 6H_2O$ (0.1) | $H_2O_2$ (2) | MeCN | 38 |
| 3 | $Cu(BF_4)_2 \cdot 6H_2O$ (0.1) | $K_2S_2O_8$ (2) | MeCN | 46 |
| 4 | $Cu(BF_4)_2 \cdot 6H_2O$ (0.1) | Oxone (2) | MeCN | 28 |
| 5 | $Cu(BF_4)_2 \cdot 6H_2O$ (0.1) | *t*-BuOOH (70% aq.) (3) | MeCN | 64 |
| 6 | $Cu(BF_4)_2 \cdot 6H_2O$ (0.1); bpy (0.1) | *t*-BuOOH (70% aq.) (3) | MeCN | 64 |
| 7[a] | $Cu(BF_4)_2 \cdot 6H_2O$ (0.1) | *t*-BuOOH (70% aq.) (3) | MeCN | 64 |
| 8 | $Cu(BF_4)_2 \cdot 6H_2O$ (0.1) | $(t\text{-BuO})_2$ (2) | MeCN | 39 |
| 9 | $Cu(BF_4)_2 \cdot 6H_2O$ (0.1) | $PhI(OAc)_2$ (2) | MeCN | Trace |
| 10 | $Cu(ClO_4)_2 \cdot 6H_2O$ (0.1) | $O_2$ | MeCN | 24 |
| 11 | $Cu(ClO_4)_2 \cdot 6H_2O$ (0.1) | $H_2O_2$ (2) | MeCN | 21 |
| 12 | $Cu(ClO_4)_2 \cdot 6H_2O$ (0.1) | $K_2S_2O_8$ (2) | MeCN-$H_2O$ (3:2 v/v) | 54 |
| 13 | $Cu(ClO_4)_2 \cdot 6H_2O$ (0.1) | $(NH_4)_2S_2O_8$ (2) | MeCN-$H_2O$ (3:2 v/v) | 56 |
| 14 | $Cu(ClO_4)_2 \cdot 6H_2O$ (0.1) | *t*-BuOOH (70% aq.) (3) | MeCN | 51 |
| 15 | $Cu(ClO_4)_2 \cdot 6H_2O$ (0.1) | *t*-BuOOH (5.0-6.0 M in decane) (3) | MeCN | 50 |
| 16 | $Cu(ClO_4)_2 \cdot 6H_2O$ (0.05) | *t*-BuOOH (70% aq.) (3) | MeCN | 45 |
| 17 | $CuCl_2$ (0.1) | *t*-BuOOH (70% aq.) (3) | MeCN | 58 |

| | | | | |
|---|---|---|---|---|
| 18 | CuI (0.1) | *t*-BuOOH (70% aq.) (3) | MeCN | 13 |
| 19 | CuCl (0.1) | $H_2O_2$ (34% aq.) (3) | MeCN | 35 |
| 20 | CuCl (0.1) | *t*-BuOOH (70% aq.) (3) | MeCN | 40 |
| 21 | $CuSO_4 \cdot 5H_2O$ (0.1) | $K_2S_2O_8$ (2) | MeCN-$H_2O$ (3:2 v/v) | 43 |
| 22 | $CuSO_4 \cdot 5H_2O$ (0.1) | *t*-BuOOH (70% aq.) (3) | MeCN | 11 |
| 23 | $Cu(OAc)_2$ (0.1) | $(NH_4)_2S_2O_8$ (2) | MeCN-$H_2O$ (3:2 v/v) | Trace |
| 24 | $Cu(OAc)_2$ (0.1) | *t*-BuOOH (70% aq.) (3) | MeCN | 27 |
| 25 | $Cu(OTf)_2$ (0.1) | *t*-BuOOH (70% aq.) (3) | MeCN | 50 |
| 26 | $(NH_4)_2CuCl_4 \cdot 2H_2O$ (0.1) | *t*-BuOOH (70% aq.) (3) | MeCN | 3 |
| 27 | $Mn(OAc)_3 \cdot 2H_2O$ (0.1) | *t*-BuOOH (70% aq.) (3) | AcOH | 26 |
| 28 | $Mn(OAc)_3 \cdot 2H_2O$ (0.1) | *t*-BuOOH (5.0-6.0 M in decane) (3) | AcOH | 45 |
| 29[b] | $Mn(OAc)_3 \cdot 2H_2O$ (0.1) | *t*-BuOOH (70% aq.) (3) | AcOH | 20 |
| 30 | $Mn(ClO_4)_2 \cdot 6H_2O$ (0.1) | *t*-BuOOH (70% aq.) (3) | MeCN | Not detected |
| 31 | $Fe(ClO_4)_3 \cdot 11H_2O$ (0.1) | *t*-BuOOH (70% aq.) (3) | MeCN | 34 |
| 32 | $Ni(OAc)_2 \cdot 4H_2O$ (0.1) | *t*-BuOOH (70% aq.) (3) | MeCN | Not detected |
| 33 | $AgNO_3$ (0.1) | $(NH_4)_2S_2O_8$ (2) | MeCN | Trace |
| 34 | *n*-$Bu_4NI$ (0.1) | *t*-BuOOH (70% aq.) (3) | MeCN | Trace |
| 35 | *n*-$Bu_4NI$ (0.1) | *t*-BuOOH (5.0-6.0 M in decane) (3) | MeCN | Trace |

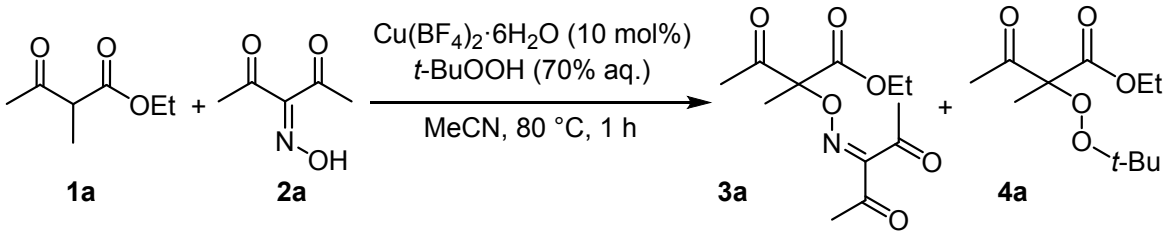
**General reaction conditions:** 2-Methylacetoacetate **1a** (144 mg, 1 mmol), 3-(hydroxyimino)pentan-2,4-dione **2a** (129 mg, 1 mmol), solvent (5 mL), and metal salt (10-55 mg, 0.1 mmol) were successively loaded into a round bottom flask. The mixture was heated on oil bath (80 °C) with stirring by a magnetic bar and an oxidant (200-615 mg, 2-3 mmol) was added for 10 seconds; stirring was continued for 1 h at 80 °C. In the entries 1 and 10 oxygen gas was bubbled through the reaction mixture (0.3 mL/sec.) until the end of the synthesis; entries 2-9, 11-35 were carried out in air atmosphere. Reaction mixture was cooled to the room temperature, diluted with $CH_2Cl_2$ (10 mL) and water (30 mL) and shaken. Organic layer was separated and aqueous layer was extracted with $CH_2Cl_2$ (3×10 mL). All the organic extracts were combined, washed with aqueous solution of $Na_2S_2O_4$ (200 mg in 20 mL of water), then with water (20 mL), dried over $Na_2SO_4$, rotary evaporated at 40-60 °C under water-jet vacuum (20-30 mmHg). C-O coupling product **3a** was isolated by column chromatography on silica gel using $CH_2Cl_2$/EtOAc eluent with volume part of EtOAc 2.5%.

[a] In the entry 7 *t*-BuOOH (70% aq.) was added dropwise for 30 min and reaction continued for an additional 30 min.

[b] In the entry 29 mixture of β-keto ester **1a** (1 mmol), oxime **2a** (1.5 mmol), $Mn(OAc)_3 \cdot 2H_2O$ (0.1 mmol, 10 mol%), *t*-BuOOH (70% aq.) (3 mmol) and MeCN (2.5 mL) was stirred at 20-25 °C for 48 h.

## 3. Determination of the influence of the molar ratio of β-keto ester 1a, oxime 2a and *t*-BuOOH on the yields of the products 3a and 4a (experiment detail for the Table 2)

**Scheme S1.**

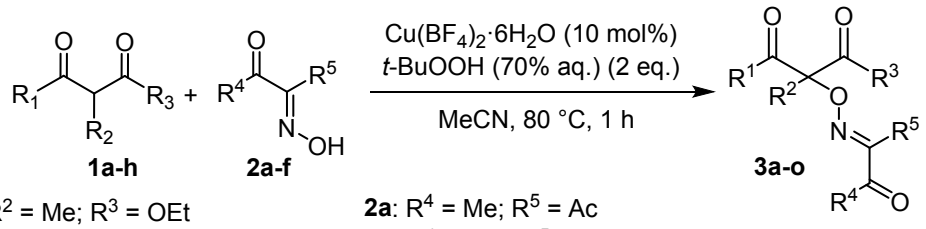



2-Methylacetoacetate **1a** (144-288 mg, 1-2 mmol), 3-(hydroxyimino)pentan-2,4-dione **2a** (129-387 mg, 1-3 mmol), MeCN (5 mL), and $Cu(BF_4)_2$•$6H_2O$ (35 mg, 0.1 mmol) were successively loaded into a round bottom flask. The mixture was heated on oil bath (80 °C) with stirring by a magnetic bar and *t*-BuOOH (70% aqueous solution, 257-386 mg, 2-3 mmol) was added for 10 seconds; stirring was continued for 1 h at 80 °C.

Reaction mixture was cooled to the room temperature, diluted with $CH_2Cl_2$ (10 mL) and water (30 mL) and shaken. Organic layer was separated and aqueous layer was extracted with $CH_2Cl_2$ (3×10 mL). All the organic extracts were combined, washed with aqueous solution of $Na_2S_2O_4$ (200 mg in 20 mL of water), then with water (20 mL), dried over $Na_2SO_4$, rotary evaporated at 40-60 °C under water-jet vacuum (20-30 mmHg). Yields of **3a** and **4a** were determined by $^1H$ NMR using *p*-methoxyacetophenone as an internal standard. In the entries 2 and 3 product **3a** was isolated by column chromatography on silica gel using $CH_2Cl_2$/EtOAc eluent with volume part of EtOAc 2.5% (isolated yields are given in parenthesis).

## 4. Oxidative C-O coupling of β-dicarbonyl compounds (experimental details for the Table 3)

**Scheme S2.**




**1a-h** **2a-f** **3a-o**

**1a**: $R^1$, $R^2$ = Me; $R^3$ = OEt
**1b**: $R^1$, $R^2$ = -$O(CH_2)_2$-; $R^3$ = Me
**1c**: $R^1$, $R^2$ = -$(CH_2)_{10}$-, $R^3$ = OEt
**1d**: $R^1$ = Me; $R^2$ = $CH_2COOEt$; $R^3$ = OEt
**1e**: $R^1$ = *i*-Pr; $R^2$ = Me; $R^3$ = OEt
**1f**: $R^1$ = Me; $R^2$ = Bn; $R^3$ = OEt
**1g**: $R^1$, $R^2$, $R^3$ = Me
**1h**: $R^1$, $R^3$ = Me; $R^2$ = Bn

**2a**: $R^4$ = Me; $R^5$ = Ac
**2b**: $R^4$ = OEt; $R^5$ = Ac
**2c**: $R^4$ = OEt, $R^5$ = COOEt
**2d**: $R^4$ = OEt; $R^5$ = Me
**2e**: $R^4$ = Ph; $R^5$ = Bz
**2f**: $R^4$ = *t*-Bu; $R^5$ = Piv

**3a**: $R^1$, $R^2$ = Me; $R^3$ = OEt**;** $R^4$ = Me; $R^5$ = Ac
**3b**: $R^1$, $R^2$ = Me; $R^3$ = OEt**;** $R^4$ = OEt; $R^5$ = Ac
**3c**: $R^1$, $R^2$ = Me; $R^3$ = OEt**;** $R^4$ = OEt, $R^5$ = COOEt
**3d**: $R^1$, $R^2$ = Me; $R^3$ = OEt**;** $R^4$ = OEt; $R^5$ = Me
**3e**: $R^1$, $R^2$ = Me; $R^3$ = OEt**;** $R^4$ = Ph; $R^5$ = Bz
**3f**: $R^1$, $R^2$ = Me; $R^3$ = OEt**;** $R^4$ = *t*-Bu; $R^5$ = Piv
**3g**: $R^1$, $R^2$ = -$O(CH_2)_2$-; $R^3$ = Me; $R^4$ = Me; $R^5$ = Ac
**3h**: $R^1$, $R^2$ = -$O(CH_2)_2$-; $R^3$ = Me; $R^4$ = OEt; $R^5$ = Ac
**3i**: $R^1$, $R^2$ = -$(CH_2)_{10}$-, $R^3$ = OEt**;** $R^4$ = Me; $R^5$ = Ac
**3j**: $R^1$ = Me; $R^2$ = $CH_2COOEt$; $R^3$ = OEt**;** $R^4$ = Me; $R^5$ = Ac
**3k**: $R^1$ = *i*-Pr; $R^2$ = Me; $R^3$ = OEt**;** $R^4$ = Me; $R^5$ = Ac
**3l**: $R^1$ = Me; $R^2$ = Bn; $R^3$ = OEt**;** $R^4$ = Me; $R^5$ = Ac
**3m**: $R^1$ = Me; $R^2$ = Bn; $R^3$ = OEt**;** $R^4$ = OEt; $R^5$ = Ac
**3n**: $R^1$, $R^2$, $R^3$ = Me**;** $R^4$ = Me; $R^5$ = Ac
**3o**: $R^1$, $R^3$ = Me; $R^2$ = Bn; $R^4$ = Me; $R^5$ = Ac

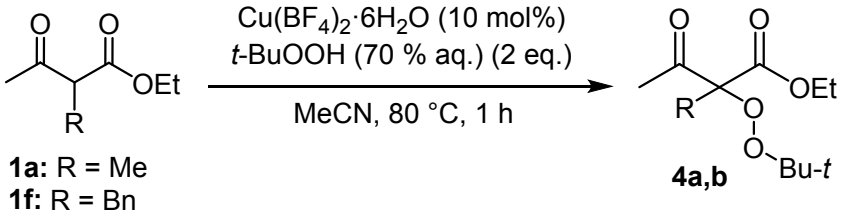
β-Dicarbonyl compound **1a-h** (114-254 mg, 1 mmol), oxime **2a-f** (194-380 mg, 1.5 mmol), MeCN (5 mL), and $Cu(BF_4)_2$•$6H_2O$ (35 mg, 0.1 mmol) were successively loaded into a round bottom flask. The mixture was heated on oil bath (80 °C) with stirring by a magnetic bar and *t*-BuOOH (70% aqueous solution, 257 mg, 2 mmol) was added for 10 seconds; stirring was continued for 1 h at 80 °C.

Reaction mixture was cooled to the room temperature, diluted with $CH_2Cl_2$ (10 mL) and water (30 mL) and shaken. Organic layer was separated and aqueous layer was extracted with $CH_2Cl_2$ (3×10 mL). All the organic extracts were combined, washed with aqueous solution of $Na_2S_2O_4$ (200 mg in 20 mL of water), then with water (20 mL), dried over $Na_2SO_4$, rotary evaporated at 40-60 °C under water-jet vacuum (20-30 mmHg). C-O coupling product **3a-o** was isolated by column chromatography on silica gel using $CH_2Cl_2$/EtOAc eluent.

## 5. Investigation into the mechanism of reaction

### 5.1 Peroxidation of β-keto esters 1a,f in the presence of Cu(II)/*t*-BuOOH (experimental details for the Scheme 4)

**Scheme S3.**




To a stirred at 80 °C mixture of β-keto ester **1a,f** (144-220 mg, 1 mmol), $Cu(BF_4)_2 \cdot 6H_2O$ (35 mg, 0.1 mmol) and MeCN (5 mL) *t*-BuOOH (70% aqueous solution, 257 mg, 2 mmol) was added for 10 seconds; stirring was continued at 80 °C for 1 h.

Reaction mixture was cooled to the room temperature, diluted with $CH_2Cl_2$ (10 mL) and water (30 mL) and shaken. Organic layer was separated and aqueous layer was extracted with $CH_2Cl_2$ (3×10 mL). All the organic extracts were combined, washed with aqueous solution of $Na_2S_2O_4$ (200 mg in 20 mL of water), then with water (20 mL), dried over $Na_2SO_4$, rotary evaporated at 40-60 °C under water-jet vacuum (20-30 mmHg). Peroxidation products **4a,b** were isolated by column chromatography on silica gel using $CH_2Cl_2$/EtOAc eluent with volume part of EtOAc 3%.

### 5.2 Cyclic voltammetry study of the redox properties of oxime 2a in the absence and presence of $Cu(BF_4)_2$

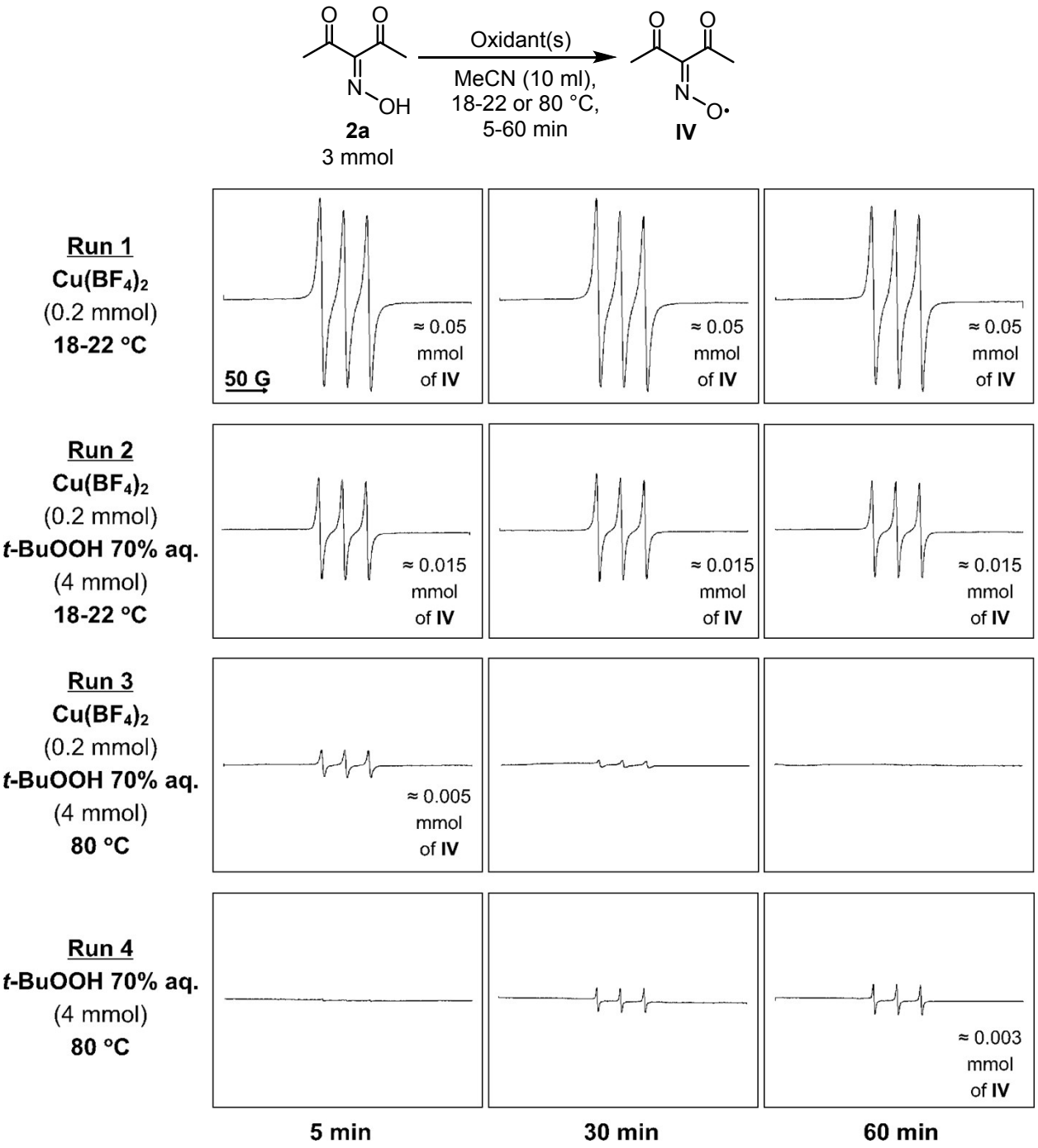
Oxime **2a** (3.22 mg, 0.025 mmol) and 0.1 M *n*-$Bu_4NBF_4$/MeCN (2.5 mL) were placed into electrochemical cell, the resultant solution was stirred by bubbling with argon at 25 °C. Then a solution of $Cu(BF_4)_2 \cdot 6H_2O$ (8.62 mg, 0.025 mmol) in 0.1 M *n*-$Bu_4NBF_4$/MeCN (2.5 mL) was added. The bubbling was stopped after 15 min and voltammetry curve was recorded.

### 5.3 EPR monitoring of formation of iminoxyl radical IV from oxime 2a under action of $Cu(BF_4)_2$ and t-BuOOH (experiment details for Figure 2)

EPR spectra were recorded at 18-22 °C using following parameters: microwave frequency — ≈ 9.6 GHz, central field — 3340 G, hf (100 kHz) field modulation amplitude — 1.0 G, microwave power — 31 mW, scan range — 300 G, receiver gain — $1.25 \times 10^4$. Sample solutions in 100-μL glass capillaries (inner diameter 1.2 mm) were placed into the EPR cavity. In all runs, EPR spectra were recorded after 5, 30 and 60 min of reaction. In runs 3 and 4 conducted at 80

°C, samples were taken from the reaction mixture at given times (5, 30 and 60 min), allowed to cool to 18-22 °C in air (about 5 min) and then EPR spectra were recorded. In all cases, EPR signal of diacetyliminoxyl radical **IV** (g = 2.0044, $a_N$ = 28.1 G)[11] was observed (Figure S1). The amount of radical **IV** formed in the reaction was estimated by double integration of its EPR spectrum. A 0.002M solution of 4-benzoyloxy-2,2,6,6-tetramethylpiperidine 1-oxyl (4-BzO-TEMPO) in MeCN was used as external concentration standard. It should be noted that addition of *t*-BuOOH 70% aq. (515 mg, 4 mmol) to a 0.002M solution of 4-BzO-TEMPO in MeCN (10 ml) did not lead to the decrease in the EPR signal intensity. This means that the decrease in intensity of EPR signal of **IV** in run 2 (with *t*-BuOOH 70% aq.) compared to that in run 1 (without *t*-BuOOH 70% aq.) is due to decrease in radical **IV** concentration and that the effect of solvent composition change on EPR sensitivity in negligible. Detailed procedures for runs 1-4 are given below.

**Figure S1**




**Run 1.** To a stirred at 18-22 °C solution of oxime **2a** (387.3 mg, 3 mmol) in MeCN (5 mL) a solution of $Cu(BF_4)_2 \cdot 6H_2O$ (69.0 mg, 0.2 mmol) in MeCN (5 mL) was added.

**Run 2.** To a stirred at 18-22 °C solution of oxime **2a** (387.3 mg, 3 mmol) in MeCN (5 mL) a solution of $Cu(BF_4)_2 \cdot 6H_2O$ (69.0 mg, 0.2 mmol) in MeCN (5 mL) was added, then *t*-BuOOH 70% aq. (515 mg, 4 mmol) was added.

**Run 3.** To a stirred at 18-22 °C solution of oxime **2a** (387.3 mg, 3 mmol) in MeCN (5 mL) a solution of $Cu(BF_4)_2 \cdot 6H_2O$ (69.0 mg, 0.2 mmol) in MeCN (5 mL) was added, then *t*-BuOOH

70% aq. (515 mg, 4 mmol) was added. Then the obtained solution was stirred on the oil bath (80 °C).

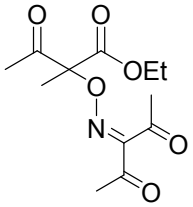
**Run 4.** To a stirred at 18-22 °C solution of oxime **2a** (387.3 mg, 3 mmol) in MeCN (5 mL) a solution of *t*-BuOOH 70% aq. (515 mg, 4 mmol) in MeCN (5 mL) was added. Then the obtained solution was stirred on the oil bath (80 °C).

### 5.4 Cyclic voltammetry study of the effect of oxime 2a and *t*-BuOOH 70% aq. on redox properties of $Cu(BF_4)_2$

Three solutions were studied by CV with scan rate 100 mV $s^{-1}$. Solution of **$Cu(BF_4)_2$**: $Cu(BF_4)_2$•$6H_2O$ (34.5 mg, 0.1 mmol) was dissolved in 0.1 M *n*-$Bu_4NBF_4$/MeCN (5 mL). Solution of **$Cu(BF_4)_2$ + Oxime 2a:** $Cu(BF_4)_2$•$6H_2O$ (34.5 mg, 0.1 mmol) was dissolved in 0.1 M *n*-$Bu_4NBF_4$/MeCN (5 mL), then oxime **2a** (193.7 mg, 1.5 mmol) was added. Solution of **$Cu(BF_4)_2$ + Oxime 2a + *t*-BuOOH:** $Cu(BF_4)_2$•$6H_2O$ (34.5 mg, 0.1 mmol) was dissolved in 0.1 M *n*-$Bu_4NBF_4$/MeCN (5 mL), then oxime **2a** (194 mg, 1.5 mmol) and *t*-BuOOH 70% aq. (257 mg, 2 mmol) were added. Solutions were deaerated by bubbling argon for 15 min before recording a cyclic voltammogram.

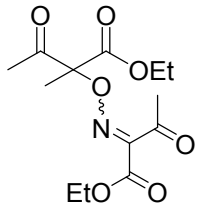
## 6. Characterization of the products

All the new compounds (**3b**, **3c**, **3e-3i**, **3k**, **3l**, **3n**, **3o** and **4a**) were characterized using $^1H$ and $^{13}C$ NMR spectroscopy, FT-IR spectroscopy, HR-MS and/or elemental analysis. $^1H$ and $^{13}C$ NMR spectra of the known compounds (**3a**, **3d**, **3j**, **3m** and **4b**) were in agreement with the literature data.[11, 12]



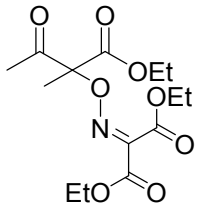

**Ethyl 2-{[(2,4-dioxopentan-3-ylidene)amino]oxy}-2-methyl-3-oxobutanoate (3a)**

Colorless oil (209 mg, 77%). **$^1H$ NMR** (300.13 MHz, $CDCl_3$) δ 4.25-4.18 (m, 2H, $OCH_2$), 2.40 (s, 3H, $CH_3$), 2.34 (s, 3H, $CH_3$), 2.23 (s, 3H, $CH_3$), 1.72 (s, 3H, $CH_3$), 1.28 (t, *J* = 7.1 Hz, 3H, $CH_3$). **$^{13}C$ NMR** (75.47 MHz, $CDCl_3$) δ 201.05, 197.2, 193.6 (C=O), 167.4 (COO), 157.3 (C=N), 91.5 (CON), 62.4 ($CH_2O$), 30.4, 25.8, 25.6, 19.3, 14.1 ($CH_2$, $CH_3$).



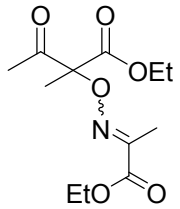
**Ethyl 2-{[(1-ethoxy-1,3-dioxobutan-2-ylidene)amino]oxy}-2-methyl-3-oxobutanoate (mixture of E and Z isomers 1:1) (3b)**

Colorless oil (105 mg, 35%). **$^1$H NMR** (300.13 MHz, $CDCl_3$) δ 4.45-4.15 (m, 4H, $OCH_2$), 2.47 (s, 1.5H, $CH_3$), 2.34 (s, 1.5H, $CH_3$), 2.23 (s, 1.5H, $CH_3$), 2.21 (s, 1.5H, $CH_3$), 1.71 (s, 3H, $CH_3$), 1.40-1.19 (m, 6H, $CH_3$). **$^{13}$C NMR** (75.47 MHz, $CDCl_3$) δ 201.5, 201.3, 195.9, 192.2 (C=O), 167.5, 167.1, 160.4, 159.7 (COO), 152.1, 151.8 (C=N), 91.8, 91.7 (CON), 62.7, 62.4 ($CH_2O$), 30.1, 25.8, 25.5, 19.3, 14.3, 14.1 ($CH_3$). **FT-IR** (KBr, thin layer) $\nu_{max}$ ($cm^{-1}$): 2987 ($CH_3$), 1752, 1732 (C=O), 1372, 1359, 1321, 1272, 1224, 1132, 1105, 1086, 1019, 981, 955. **HRMS** (ESI) *m*/*z* calc. for $C_{13}H_{19}NO_7$, $[M+Na]^+$: 324.1054; Found 324.1051.



**Diethyl 2-{[(1-ethoxy-2-methyl-1,3-dioxobutan-2-yl)oxy]imino}malonate (3c)**

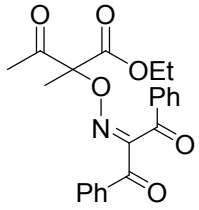
Colorless oil (179 mg, 54%). **$^1$H NMR** (300.13 MHz, $CDCl_3$) δ 4.41 (q, *J* = 7.2 Hz, 2H, $OCH_2$), 4.33 (q, *J* = 7.1 Hz, 2H, $OCH_2$), 4.29-4.16 (m, 2H, $OCH_2$), 2.24 (s, 3H, $CH_3$), 1.72 (s, 3H, $CH_3$), 1.40-1.24 (m, 9H, $CH_3$). **$^{13}$C NMR** (75.47 MHz, $CDCl_3$) δ 201.8 (C=O), 167.1, 160.0, 159.2 (COO), 146.0 (C=N), 91.9 (CON), 62.8, 62.6, 62.4 ($CH_2O$), 25.8, 19.3, 14.2, 14.1 ($CH_3$). **FT-IR** (KBr, thin layer) $\nu_{max}$ ($cm^{-1}$): 2987, 2943, 2910 ($CH_2$, $CH_3$), 1752 (C=O), 1468, 1447, 1394, 1373, 1329, 1298, 1259, 1222, 1174, 1132, 1093, 1017, 983, 860. **HRMS** (ESI) *m*/*z* calc. for $C_{14}H_{21}NO_8$, $[M+Na]^+$: 354.1161; Found 354.1159. **Elemental analysis** found: C, 50.65; H, 6.38; N, 4.25. Calc. for $C_{14}H_{21}NO_8$: C 50.75, H 6.39, N 4.23%.



**Ethyl 2-{[(1-ethoxy-1-oxopropan-2-ylidene)amino]oxy}-2-methyl-3-oxobutanoate (mixture of *E* and *Z* isomers 10:1) (3d)**

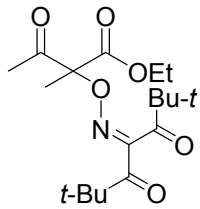
Colorless oil (104 mg, 38%). **$^1$H NMR** (300.13 MHz, $CDCl_3$) δ 4.38-4.12 (m, 4H, $OCH_2$), 2.25 (s, 2.87H, $CH_3$), 2.22 (s, 0.7H, $CH_3$), 2.15 (s, 2.73H, $CH_3$), 2.05 (s, 0.27H, $CH_3$), 1.68 (s, 2.73H,

$CH_3$), 1.61 (s, 0.27H, $CH_3$), 1.40-1.18 (m, 6H, $CH_3$). **$^{13}$C NMR** (75.47 MHz, $CDCl_3$) δ 202.8 (C=O), 168.2, 163.5 (COO), 151.4 (C=N), 90.8 (CON), 62.1, 61.9 ($CH_2O$), 25.9, 19.7, 14.2, 14.1, 11.9 ($CH_3$).



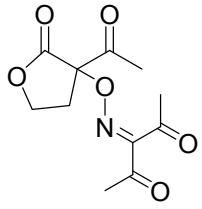
**Ethyl 2-{[(1,3-dioxo-1,3-diphenylpropan-2-ylidene)amino]oxy}-2-methyl-3-oxobutanoate (3e)**

Colorless oil (158 mg, 40%). **$^{1}$H NMR** (300.13 MHz, $CDCl_3$) δ 8.13-8.06 (m, 2H, ArH), 7.71-7.59 (m 2H, ArH), 7.59-7.43 (m, 2H, ArH), 4.22 (q, $J$ = 7.1 Hz, 2H, $OCH_2$), 1.96 (s, 3H, $CH_3$), 1.66 (s, 3H, $CH_3$), 1.24 (t, $J$ = 7.1 Hz, 3H, $CH_3$). **$^{13}$C NMR** (75.47 MHz, $CDCl_3$) δ 201.9, 190.5, 187.5 (C=O), 167.7 (COO), 156.0 (C=N), 135.3, 134.9, 134.6, 134.3, 130.7, 129.5, 129.1, 128.6 ($C_{Ar}$), 91.8 (CON), 62.4 ($CH_2O$), 25.6, 19.6, 14.1 ($CH_3$). **FT-IR** (KBr, thin layer) $\nu_{max}$ ($cm^{-1}$): 1752, 1731, 1689, 1657 (C=O), 1597, 1450, 1325, 1264, 1116, 945. **Elemental analysis** found: 66.79, H, 5.41, N, 3.76. Calc. for $C_{22}H_{21}NO_6$: C 66.83, H 5.35, N 3.54 %.



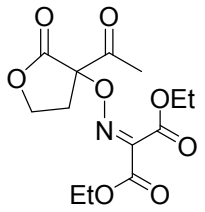
**Ethyl 2-methyl-3-oxo-2-{[(2,2,6,6-tetramethyl-3,5-dioxoheptan-4-ylidene)amino]oxy}butanoate (3f)**

Colorless oil (89 mg, 25%). **$^{1}$H NMR** (300.13 MHz, $CDCl_3$) δ 4.23 (q, $J$ = 7.1 Hz, 2H, $OCH_2$), 2.22 (s, 3H, $CH_3$), 1.70 (s, 3H, $CH_3$), 1.27 (t, $J$ = 7.1 Hz, 3H, $CH_3$), 1.26 (s, 9H, $3CH_3$), 1.20 (s, 9H, $CH_3$). **$^{13}$C NMR** (75.47 MHz, $CDCl_3$) δ 208.4, 201.7, 201.5 (C=O), 167.7 (COO), 156.7 (C=N), 91.2 (CON), 62.5 ($CH_2O$), 45.0 ($\underline{C}(CH_3)_3$), 43.6 ($\underline{C}(CH_3)_3$), 27.0, 26.3, 25.7, 19.8, 14.1 ($CH_3$). **FT-IR** (KBr, thin layer) $\nu_{max}$ ($cm^{-1}$): 2977 ($CH_3$), 1754, 1734, 1716 (C=O), 1677 1840, 1366, 1269, 1125, 937. **HRMS** (ESI) $m/z$ calc. for $C_{18}H_{29}NO_6$, $[M+Na]^+$: 378.1887; Found 378.1887.



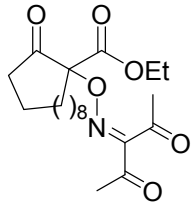
**3-{[(3-acetyl-2-oxotetrahydrofuran-3-yl)oxy]imino}pentane-2,4-dione (3g)**

Colorless oil (166 mg, 65%). **$^1$H NMR** (300.13 MHz, $CDCl_3$) δ 4.55-4.37 (m, 2H, $OCH_2$), 3.02-2.90 (m, 1H, $CH_2$), 2.70-2.53 (m, 1H, $CH_2$), 2.43 (s, 3H, $CH_3$), 2.39 (s, 3H, $CH_3$) 2.37 (s, 3H, $CH_3$). **$^{13}$C NMR** (75.47 MHz, $CDCl_3$) δ 200.6, 196.7, 193.2 (C=O), 169.4 (COO), 158.5 (C=N), 91.3 (CON), 66.1 ($CH_2O$), 30.7, 30.6, 26.1, 26.0 ($CH_2$, $CH_3$). **FT-IR** (KBr, thin layer) $\nu_{max}$ ($cm^{-1}$): 2928 ($CH_2$, $CH_3$), 1782, 1726, 1696 (C=O), 1421, 1362, 1295, 1216, 1187, 1056, 1024, 984, 935. **HRMS** (ESI) *m/z* calc. for $C_{11}H_{13}NO_6$, $[M+Na]^+$: 278.0635; Found 278.0635.



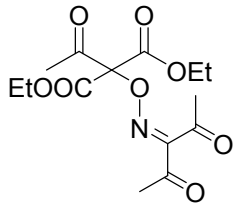

**Diethyl 2-{[(3-acetyl-2-oxotetrahydrofuran-3-yl)oxy]imino}malonate (3h)**

Colorless oil (158 mg, 50%). **$^1$H NMR** (300.13 MHz, $CDCl_3$) δ 4.54-4.26 (m, 6H, $OCH_2$), 3.06-2.93 (m, 1H, $CH_2$), 2.66-2.51 (m, 1H, $CH_2$), 2.36 (s, 3H, $CH_3$), 1.37 (t, $J$ = 7.1 Hz, $CH_3$), 1.33 (t, $J$ = 7.1 Hz, $CH_3$). **$^{13}$C NMR** (75.47 MHz, $CDCl_3$) δ 201.3 (C=O), 169.0, 159.5, 158.7 (COO), 147.5 (C=N), 91.3 (CON), 66.4, 63.2, 63.0 ($OCH_2$), 30.9, 26.2, 14.2, 14.1 ($CH_2$, $CH_3$). **FT-IR** (KBr, thin layer) $\nu_{max}$ ($cm^{-1}$): 2988, 2941 ($CH_2$, $CH_3$), 1788, 1749, 1726 (C=O), 1376, 1330, 1300, 1259, 1095, 1063, 1021, 988. **HRMS** (ESI) *m/z* calc. for $C_{14}H_{21}NO_8$, $[M+Na]^+$: 338.0846; Found 338.0853. **Elemental analysis** found: C, 49.30; H, 5.42; N, 4.46. Calc. for $C_{14}H_{21}NO_8$: C, 49.53; H, 5.44; N, 4.44%.



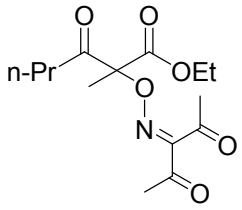

**Ethyl 1-{[(2,4-dioxopentan-3-ylidene)amino]oxy}-2-oxocyclododecanecarboxylate (3i)**

Colorless oil (198 mg, 52%). **$^1$H NMR** (300.13 MHz, $CDCl_3$) δ 4.33-4.16 (m, 2H, $OCH_2$), 2.81-1.51 (m, 2H, $CH_2$), 2.42 (s, 3H, $CH_3$), 2.39-2.08 (m, 2H, $CH_2$), 2.31 (s, 3H, $CH_3$), 1.96-1.76 (m, 1H, $CH_2$), 1.72-1.54 (m, 1H, $CH_2$), 1.45-1.13 (m, 17H, $CH_2$, $CH_3$). **$^{13}$C NMR** (75.47 MHz, $CDCl_3$) δ 202.9, 197.6, 193.9 (C=O), 168.0 (COO), 156.9 (C=N), 94.8 (CON), 62.2 ($CH_2O$), 35.5, 32.1, 30.5, 26.8, 26.3, 25.9, 23.8, 23.1, 23.0, 22.7, 21.3, 19.7, 14.2 ($CH_2$, $CH_3$). **FT-IR** (KBr, thin layer) $\nu_{max}$ ($cm^{-1}$): 2935, 2866 ($CH_2$, $CH_3$), 1751, 1728, 1694 (C=O), 1470, 1364, 1297, 1279, 1256, 1243, 1024, 982, 956. **HRMS** (ESI) *m/z* calc. for $C_{20}H_{31}NO_6$, $[M+Na]^+$: 404.2044; Found 404.2039. **Elemental analysis** found: C, 62.93; H, 8.23; N 3.71. Calc. for $C_{20}H_{31}NO_6$: C, 62.97; H, 8.19; N, 3.67%.



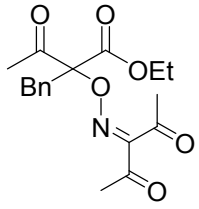
**Diethyl 2-acetyl-2-{[(2,4-dioxopentan-3-ylidene)amino]oxy}malonate (3j)**

Colorless oil (86 mg, 25%). **$^1$H NMR** (300.13 MHz, $CDCl_3$) 4.25 (q, *J* = 7.0 Hz, 2H, $OCH_2$), 4.10 (q, *J* = 7.1 Hz, 2H, $OCH_2$), 3.47 (d, *J* = 17.2 Hz, 1H, $CH_2$), 3.32 (d, *J* = 17.2 Hz, 1H, $CH_2$), 2.39 (s, 3H, $CH_3$), 2.332 (s, 3H, $CH_3$), 2.326 (s, 3H, $CH_3$), 1.35–1.13 (m, 6H, $CH_3$). **$^{13}$C NMR** (75.47 MHz, $CDCl_3$) δ 200.9, 196.9, 193.5 (C=O), 168.7, 165.7 (COO), 157.8 (C=N), 91.7 (CON), 62.9, 61.4 ($OCH_2$), 38.6, 30.5, 26.5, 26.0, 14.2, 14.1 ($CH_3$, $CH_2$).



**Ethyl 2-{[(2,4-dioxopentan-3-ylidene)amino]oxy}-2-methyl-3-oxohexanoate (3k)**

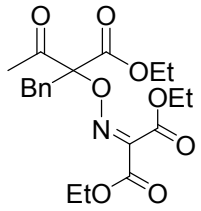
Colorless oil (161 mg, 54%). **$^1$H NMR** (300.13 MHz, $CDCl_3$) δ 4.33-4.16 (m, 2H, $OCH_2$), 2.52 (t, *J* = 7.3 Hz, 2H, $CH_2$), 2.40 (s, 3H, $CH_3$), 2.34 (s, 3H, $CH_3$), 1.72 (s, 3H, $CH_3$), 1.69-1.50 (m, 2H, $CH_2$), 1.27 (t, *J* = 7.1 Hz, 3H, $CH_3$), 0.90 (t, *J* = 7.4 Hz, 3H, $CH_3$). **$^{13}$C NMR** (75.47 MHz, $CDCl_3$) δ 203.4, 197.4, 193.8 (C=O), 167.7 (COO), 157.4 (C=N), 91.7 (CON), 62.4 ($OCH_2$), 39.7, 30.5, 25.9, 19.5, 16.8, 14.2, 13.7 ($CH_3$, $CH_2$). **FT-IR** (KBr, thin layer) $\nu_{max}$ ($cm^{-1}$): 2696, 2940 ($CH_2$, $CH_3$), 1754, 1728, 1695 (C=O), 1365, 1296, 1268, 1129, 961. **Elemental analysis** found: C, 56.03; H, 7.03; N, 4.69. Calc. for $C_{14}H_{21}NO_6$: C, 56.18; H, 7.07; N, 4.68%.



**Ethyl 2-benzyl-2-{[(2,4-dioxopentan-3-ylidene)amino]oxy}-3-oxobutanoate (3l)**

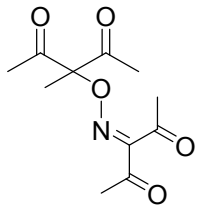
White powder (153 mg, 44%); mp = 70-71 °C. **$^1$H NMR** (300.13 MHz, $CDCl_3$) δ 7.34-7.20 (m, 3H, ArH), 7.18-7.07 (m, 2H, ArH), 4.35-4.15 (m, 2H, $OCH_2$), 3.56 (s, 2H, $CH_2$), 2.39 (s, 3H, $CH_3$), 2.24 (s, 3H, $CH_3$), 2.05 (s, 3H, $CH_3$), 1.26 (t, *J* = 7.1 Hz, 3H, $CH_3$). **$^{13}$C NMR** (75.47 MHz, $CDCl_3$) δ 201.5, 197.2, 193.6 (C=O), 166.6 (COO), 157.5 (C=N), 133.7, 130.4, 128.6, 127.6 ($C_{Ar}$), 94.3 (CON), 62.5 ($OCH_2$), 39.0 ($\underline{C}H_2Ph$), 30.1, 27.1, 26.0, 14.2 ($CH_3$). **FT-IR** (KBr, thin layer) $\nu_{max}$ ($cm^{-1}$): 3604, 3047, 3007, 2979, 2926 ($CH_2$, $CH_3$), 1756, 1731, 1721, 1693

(C=O), 1355, 1298, 1239, 1186, 938. **Elemental analysis** found: C, 61.90; H, 6.17; N, 4.08. Calc. for $C_{18}H_{21}NO_6$: C, 62.24; H, 6.09; N, 4.03%.



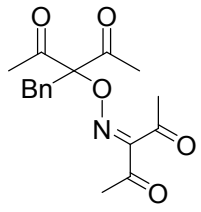
**Ethyl 2-benzyl-2-{[(2,4-dioxopentan-3-ylidene)amino]oxy}-3-oxobutanoate (3m)**

Pale yellow oil (147 mg, 36%). **$^1$H NMR** (300.13 MHz, $CDCl_3$) δ 7.34-7.22 (m, 3H, ArH), 7.18-7.08 (m, 2H, ArH), 4.47-4.13 (m, 6H, $CH_2$), 3.59 (s, 2H, $PhCH_2$), 1.98 (s, 3H, $CH_3$), 1.39 (t, *J* = 7.1 Hz, 3H, $CH_3$), 1.31 (t, *J* = 7.1 Hz, 3H), 1.25 (t, *J* = 7.1 Hz, 3H). **$^{13}$C NMR** (75.47 MHz, $CDCl_3$) δ 202.1 (C=O), 166.2, 160.0, 159.2 (COO), 146.3 (C=N), 133.9, 130.6, 128.3, 127.2 ($C_{Ar}$), 94.8 (CON), 62.9, 62.6, 62.4 ($OCH_2$), 38.9 ($CH_2Ph$), 27.2, 14.14, 14.10, 14.0 ($CH_3$).



**3-{[(2,4-dioxopentan-3-ylidene)amino]oxy}-3-methylpentane-2,4-dione (3n)**

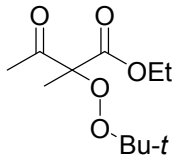
Colorless oil (123 mg, 51%). **$^1$H NMR** (300.13 MHz, $CDCl_3$) δ 2.41 (s, 3H, $CH_3$), 2.33 (s, 3H, $CH_3$), 2.19 (s, 3H, $CH_3$), 1.67 (s, 3H, $CH_3$). **$^{13}$C NMR** (75.47 MHz, $CDCl_3$) δ 201.95, 197.5, 193.7 (C=O), 157.8 (C=N), 96.7 (CON), 30.5, 26.2, 25.9, 19.2 ($CH_3$). **FT-IR** (KBr, thin layer) $\nu_{max}$ ($cm^{-1}$): 3006, 2930 ($CH_3$), 1721, 1695 (C=O), 1421, 1360, 1297, 1245, 1195, 1119, 1102, 1067, 967, 945. **Elemental analysis** found: C, 54.67; H, 6.23; N 5.80. Calc. for $C_{11}H_{15}NO_5$: C, 54.77; H, 6.27; N, 5.81%.



**3-benzyl-3-{[(2,4-dioxopentan-3-ylidene)amino]oxy}pentane-2,4-dione (3o)**

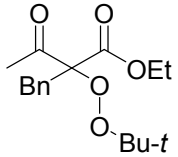
Pale yellow powder (79 mg, 25%); mp = 77-78 °C. **$^1$H NMR** (300.13 MHz, $CDCl_3$) δ 7.32-7.22 (m, 3H, ArH), 7.08-6.99 (m, 2H, ArH), 3.52 (s, 2H, $CH_2$), 2.41 (s, 3H, $CH_3$), 2.27 (s, 3H, $CH_3$), 2.03 (s, 6H, $CH_3$). **$^{13}$C NMR** (75.47 MHz, $CDCl_3$) δ 201.4, 197.3, 193.6 (C=O), 158.0 (C=N), 133.8, 130.2, 128.7, 127.6 ($C_{Ar}$), 99.5 (CON), 39.1 ($CH_2$), 30.1, 27.4, 26.0 ($CH_3$). **FT-IR** (KBr, thin layer) $\nu_{max}$ ($cm^{-1}$): 1739, 1719, 1693 (C=O), 1418, 1355, 1296, 1189, 974, 962, 941, 706,

563, 506. **Elemental analysis** found: C, 64.29; H, 6.22; N, 4.31. Calc. for $C_{17}H_{19}NO_5$: C, 64.34; H, 6.04; N, 4.41%.




**Ethyl 2-(*tert*-butylperoxy)-2-methyl-3-oxobutanoate (4a)**

Pale yellow oil (109 mg, 47%). **$^1$H NMR** (300.13 MHz, $CDCl_3$) 4.20 (q, $J$ = 7.1 Hz, 2H, $OCH_2$), 2.27 (s, 3H, $CH_3$), 1.56 (s, 3H, $CH_3$), 1.25 (t, $J$ = 7.1 Hz, 3H, $CH_3$), 1.24 (s, 9H, *t*-Bu). **$^{13}$C NMR** (75.47 MHz, $CDCl_3$) δ 203.8 (C=O), 168.15 (COO), 89.8 (COO*t*-Bu), 80.8 ($C(CH_3)_3$), 61.8 ($CH_2O$), 26.5, 25.7, 18.4, 14.1 ($CH_3$). **FT-IR** (KBr, thin layer) $\nu_{max}$ ($cm^{-1}$): 2983, 2940 ($CH_2$, $CH_3$), 1754, 1733 (C=O), 1366, 1261, 1196, 1150, 1132, 1109, 1021. **HRMS** (ESI) *m*/*z* calc. for $C_{11}H_{20}O_5$, $[M+Na]^+$: 255.1203; Found 255.1199.




**Ethyl 2-(*tert*-butylperoxy)-2-benzyl-3-oxobutanoate (4b)**

Pale yellow oil (126 mg, 41%). **$^1$H NMR** (300.13 MHz, $CDCl_3$) δ 7.28-7.14 (m, 5H, ArH), 4.28-4.08 (m, 2H, $OCH_2$), 3.57 (d, $J$ = 14.3 Hz, 1H, $CH_2Ph$), 3.34 (d, $J$ = 14.3 Hz, 1H, $CH_2Ph$), 1.90 (s, 3H, $CH_3$), 1.32 (s, 9H, *t*-Bu), 1.23 (t, $J$ = 7.1 Hz, 3H, $CH_3$). **$^{13}$C NMR** (75.47 MHz, $CDCl_3$) δ 203.7 (C=O), 167.8 (COOt), 135.1, 130.8, 128.1, 126.9 ($C_{Ar}$), 92.8 (COO*t*-Bu), 81.3 ($C(CH_3)_3$), 61.8 ($CH_2O$), 37.3 ($CH_2Ph$), 27.3, 26.7, 14.1 ($CH_3$).

**References:**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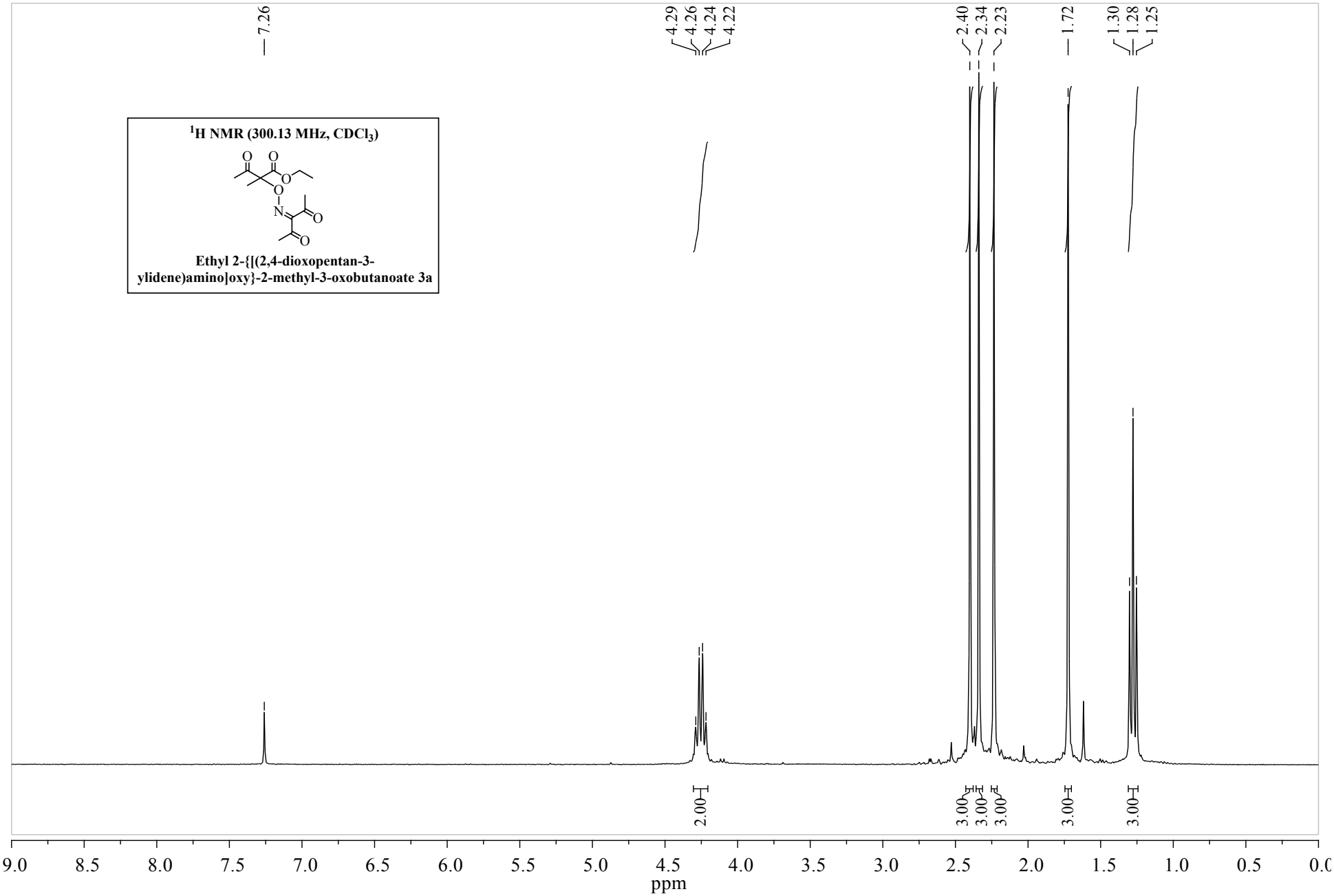
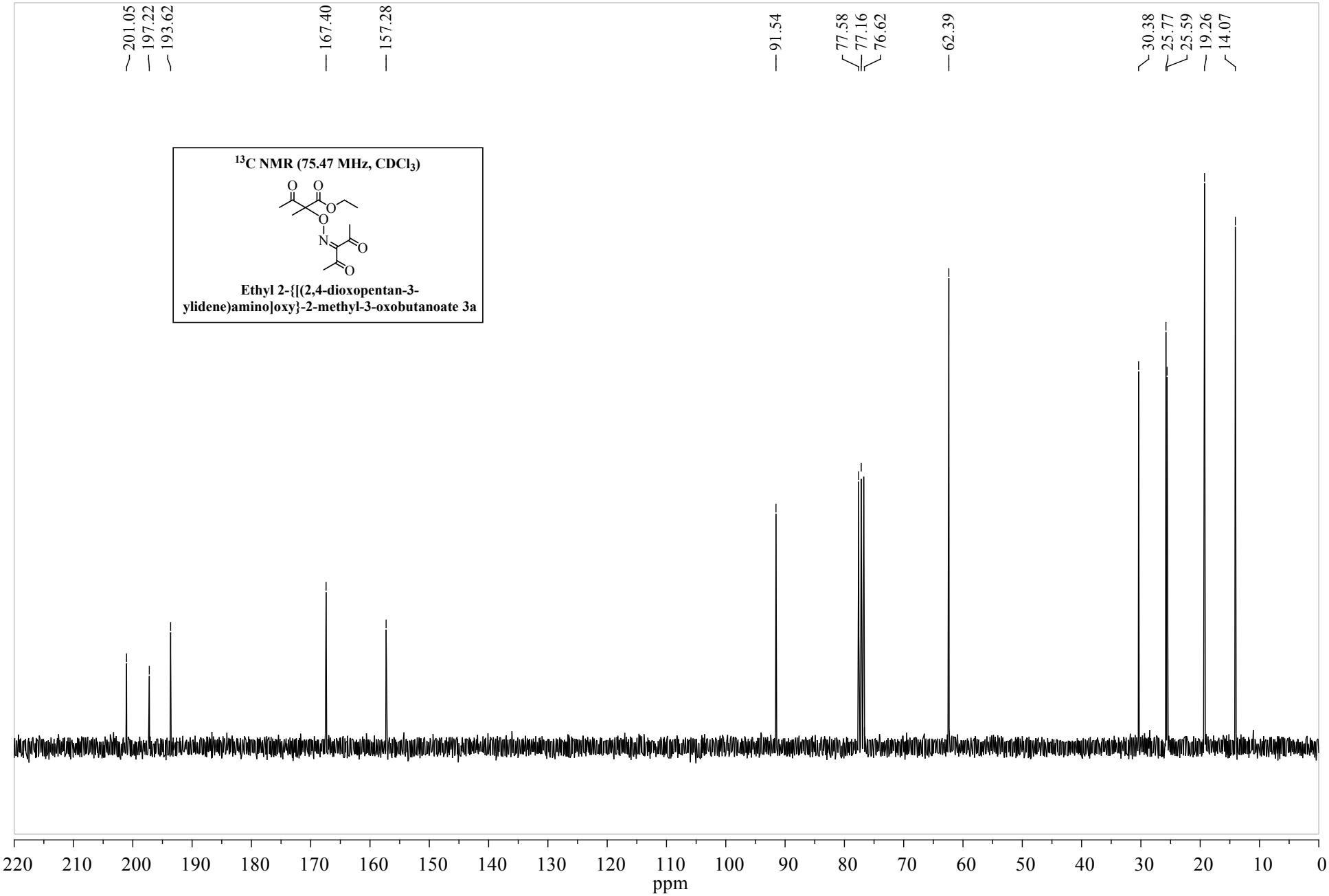
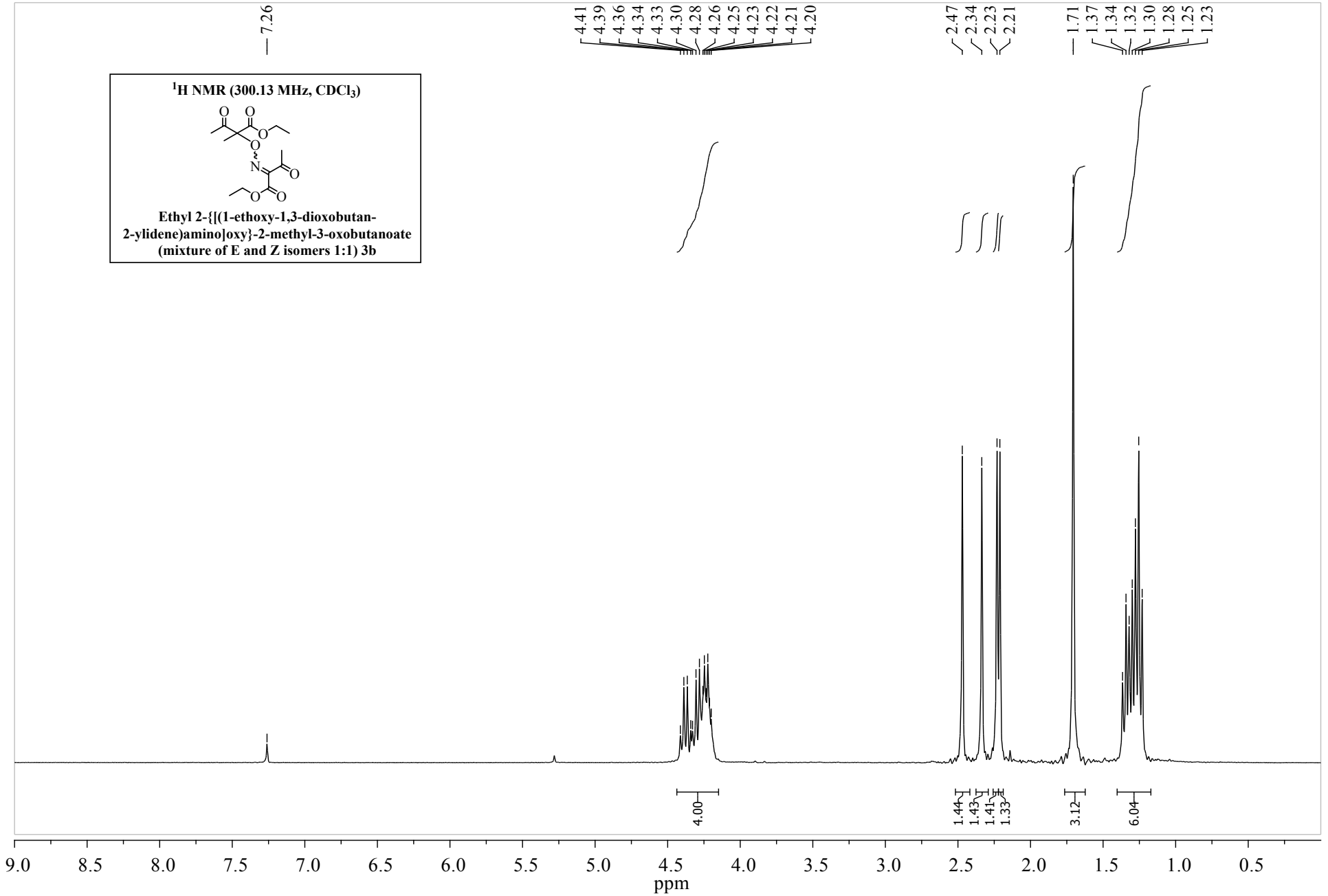
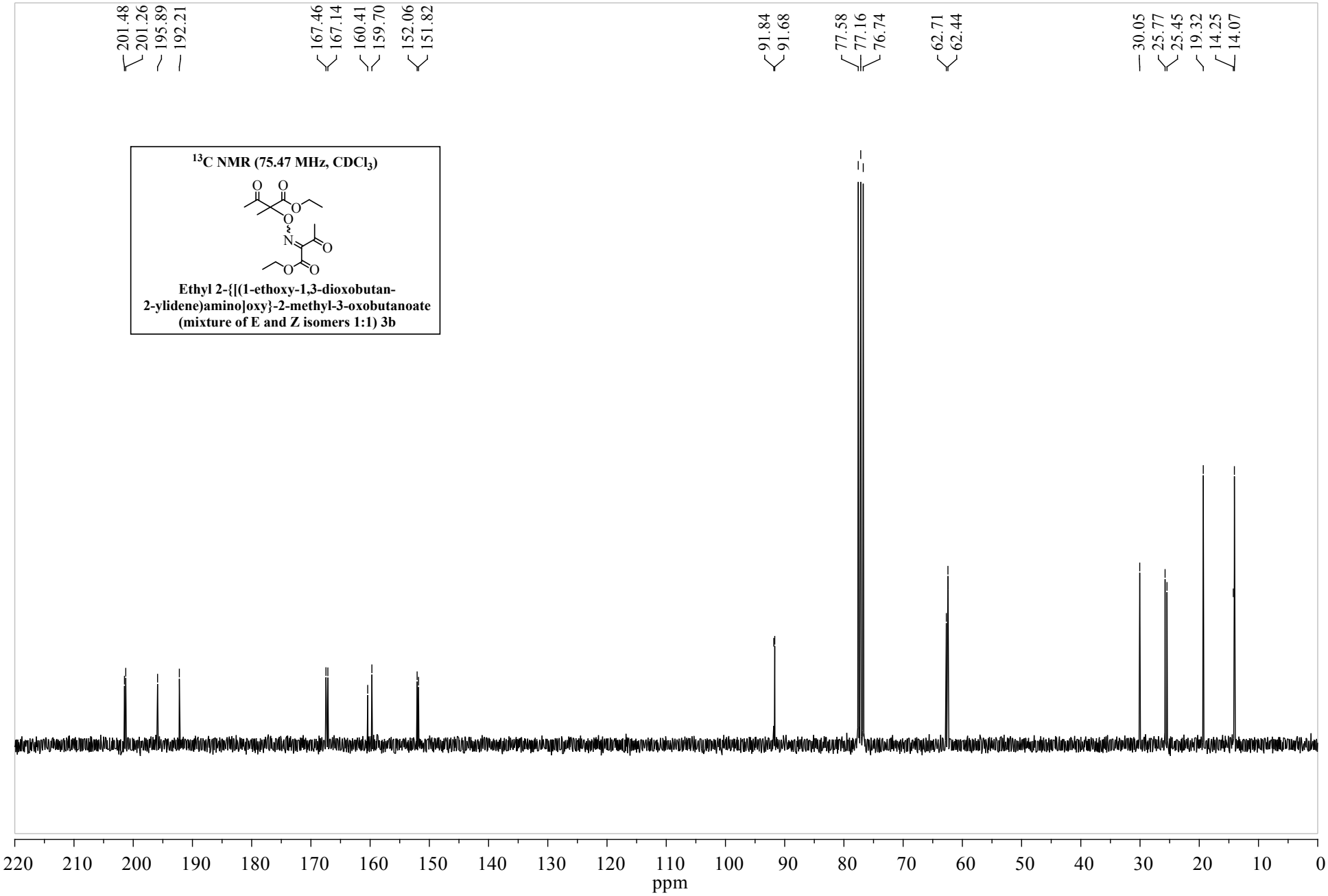
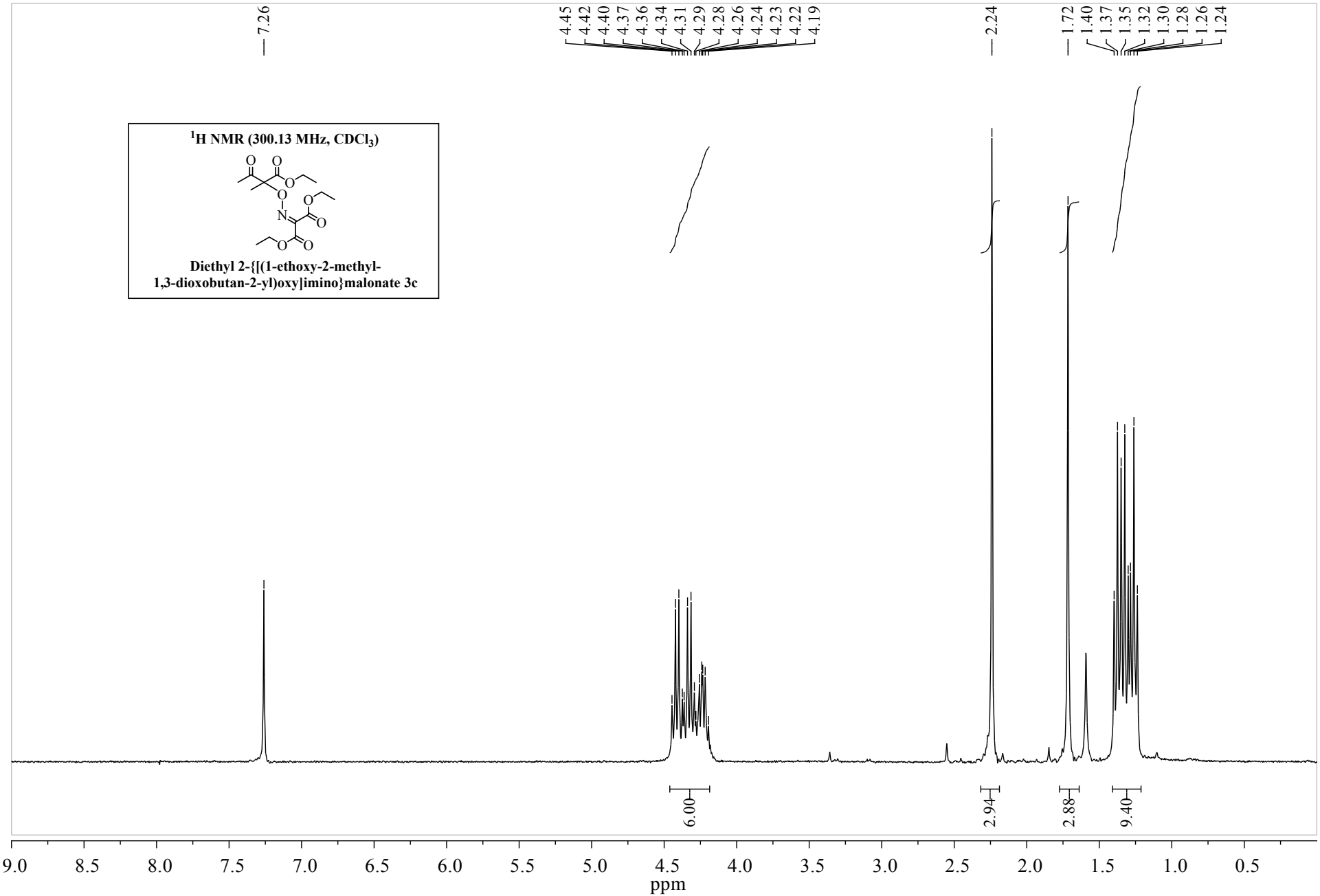
## 7. Copies of $^{1}H$, $^{13}C$ and FT-IR Spectra of synthesized products 3a-o and 4a,b

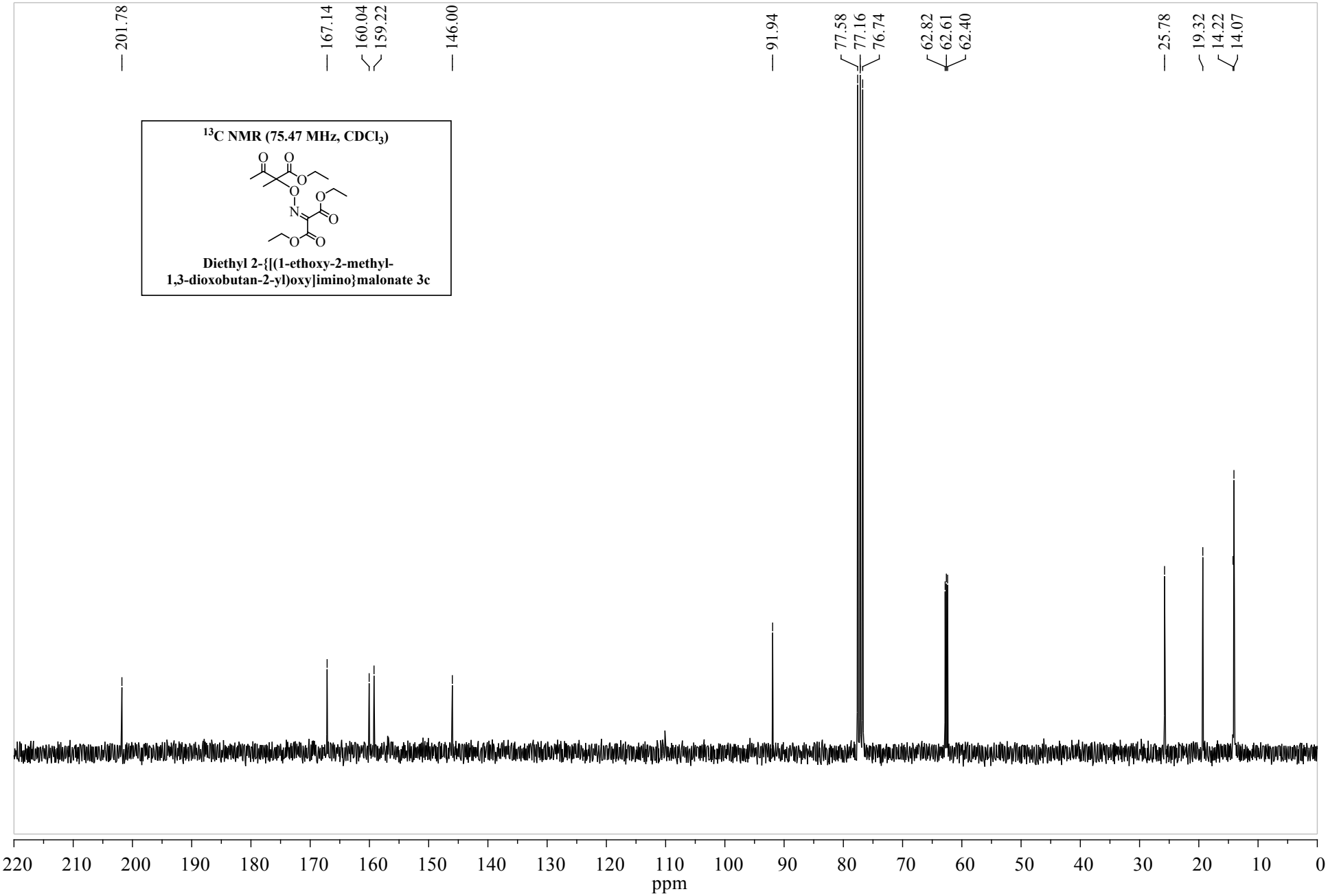

7.26
4.29
4.26
4.24
4.22
2.40
2.34
2.23
1.72
1.30
1.28
1.25
$^1$H NMR (300.13 MHz, $CDCl_3$)
Ethyl 2-{[(2,4-dioxopentan-3-ylidene)amino]oxy}-2-methyl-3-oxobutanoate 3a
2.00
3.00
3.00
3.00
3.00
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

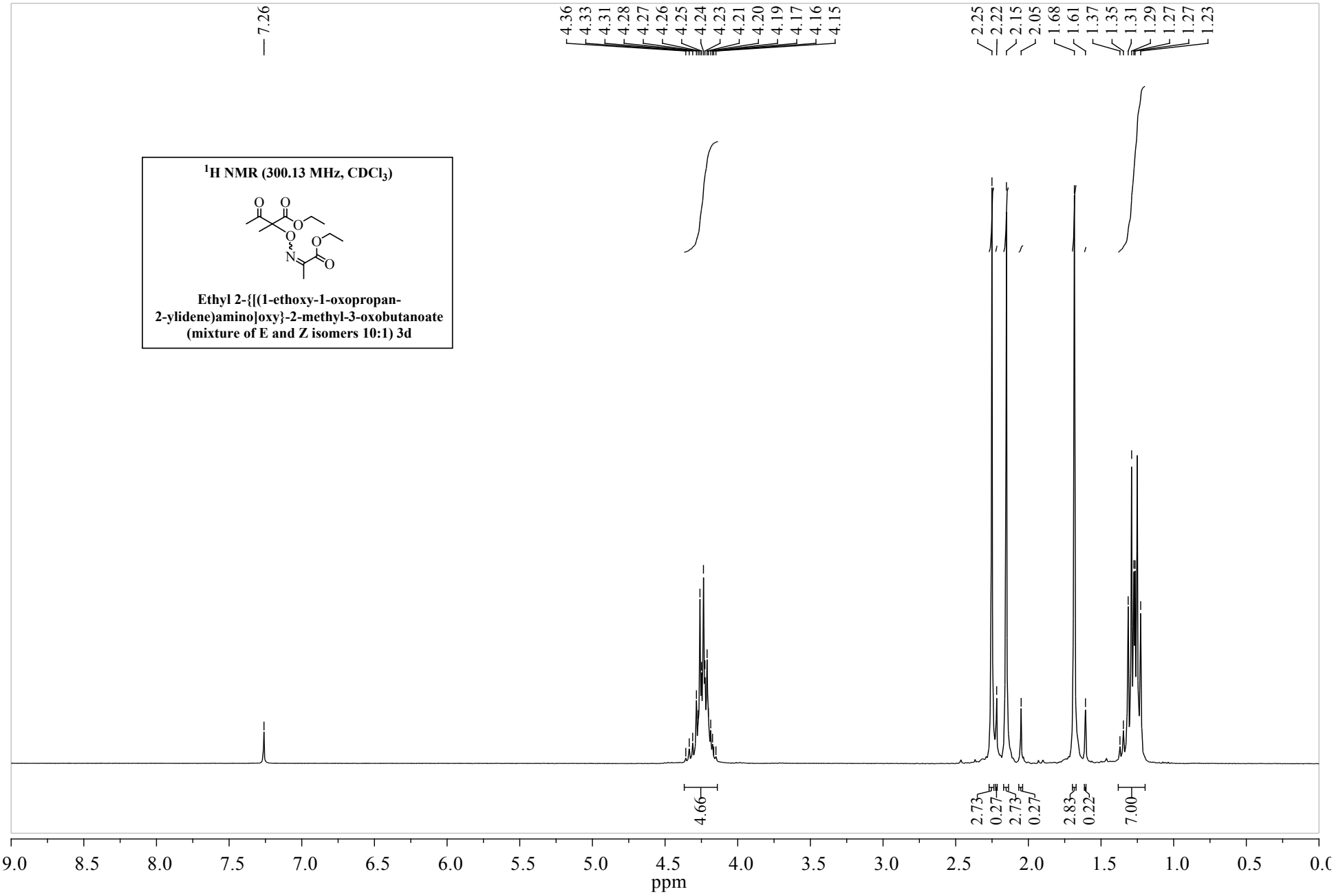

201.05
197.22
193.62
167.40
157.28
91.54
77.58
77.16
76.62
62.39
30.38
25.77
25.59
19.26
14.07
$^{13}$C NMR (75.47 MHz, $CDCl_3$)
Ethyl 2-{[(2,4-dioxopentan-3-ylidene)amino]oxy}-2-methyl-3-oxobutanoate 3a
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

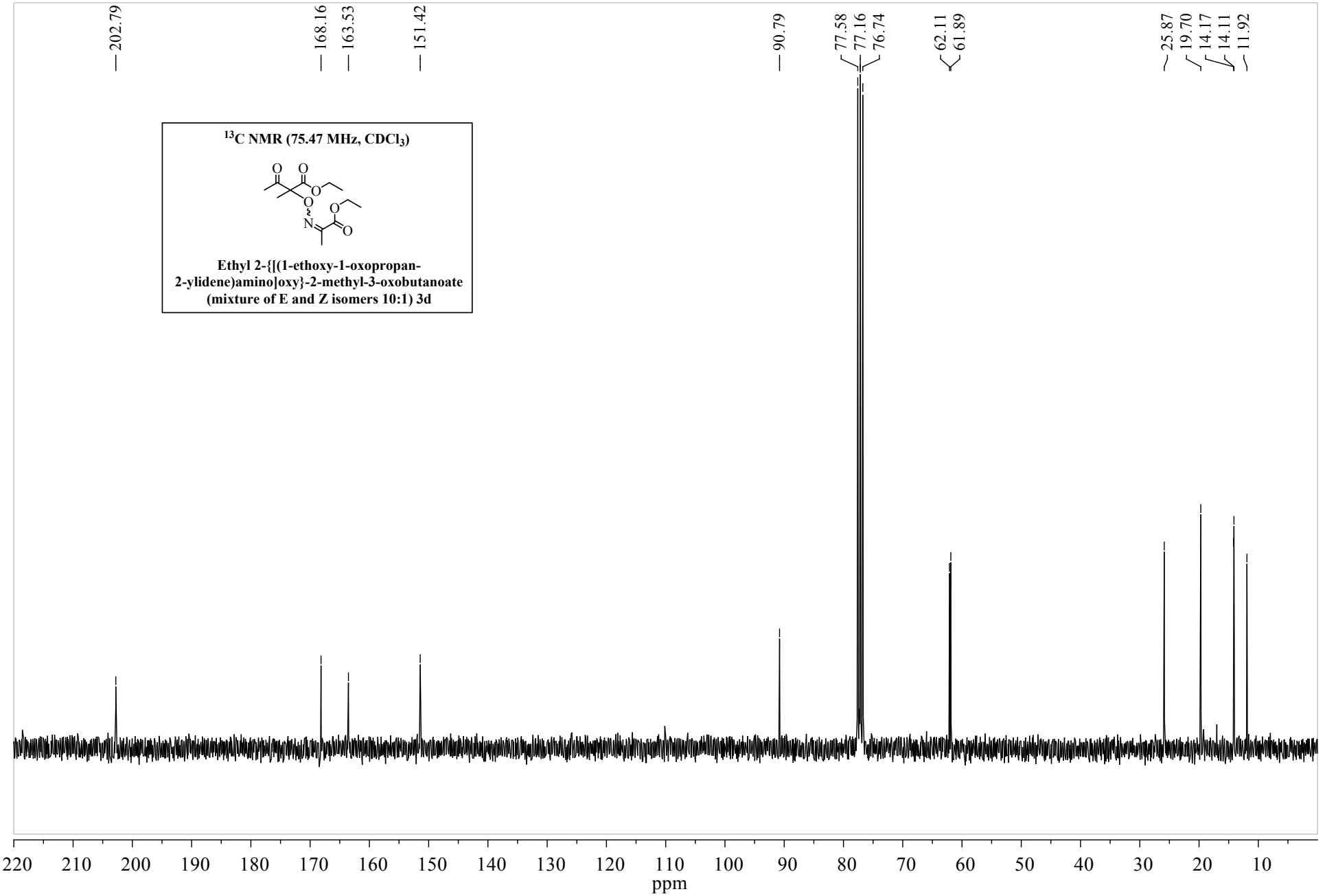

7.26
4.41
4.39
4.36
4.34
4.33
4.30
4.28
4.26
4.25
4.23
4.22
4.21
4.20
2.47
2.34
2.23
2.21
1.71
1.37
1.34
1.32
1.30
1.28
1.25
1.23
$^1$H NMR (300.13 MHz, $CDCl_3$)
Ethyl 2-{[(1-ethoxy-1,3-dioxobutan-2-ylidene)amino]oxy}-2-methyl-3-oxobutanoate (mixture of E and Z isomers 1:1) 3b
4.00
1.44
1.43
1.41
1.33
3.12
6.04
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

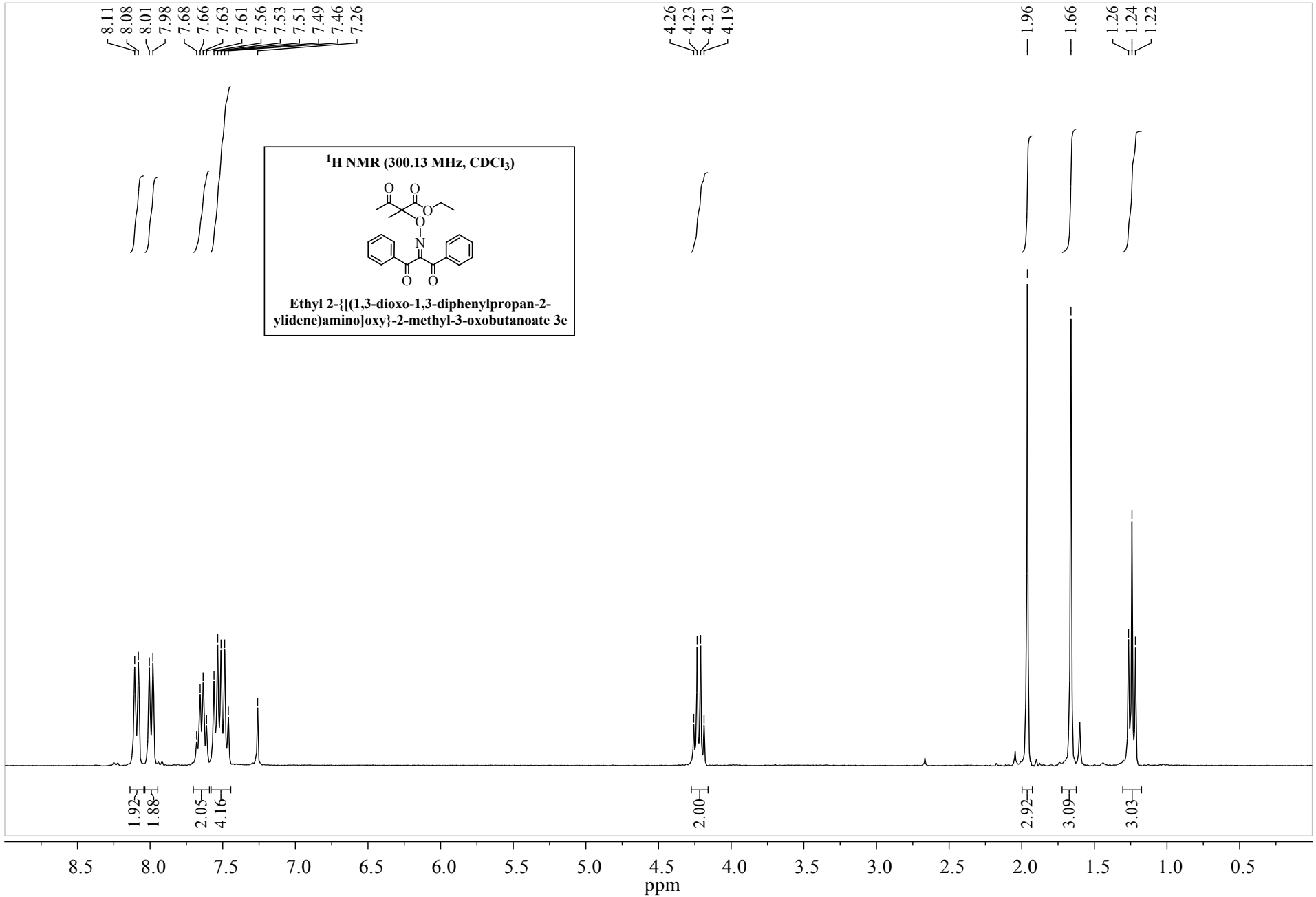

201.48
201.26
195.89
192.21
167.46
167.14
160.41
159.70
152.06
151.82
91.84
91.68
77.58
77.16
76.74
62.71
62.44
30.05
25.77
25.45
19.32
14.25
14.07
$^{13}$C NMR (75.47 MHz, $CDCl_3$)
Ethyl 2-{[(1-ethoxy-1,3-dioxobutan-2-ylidene)amino]oxy}-2-methyl-3-oxobutanoate (mixture of E and Z isomers 1:1) 3b
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

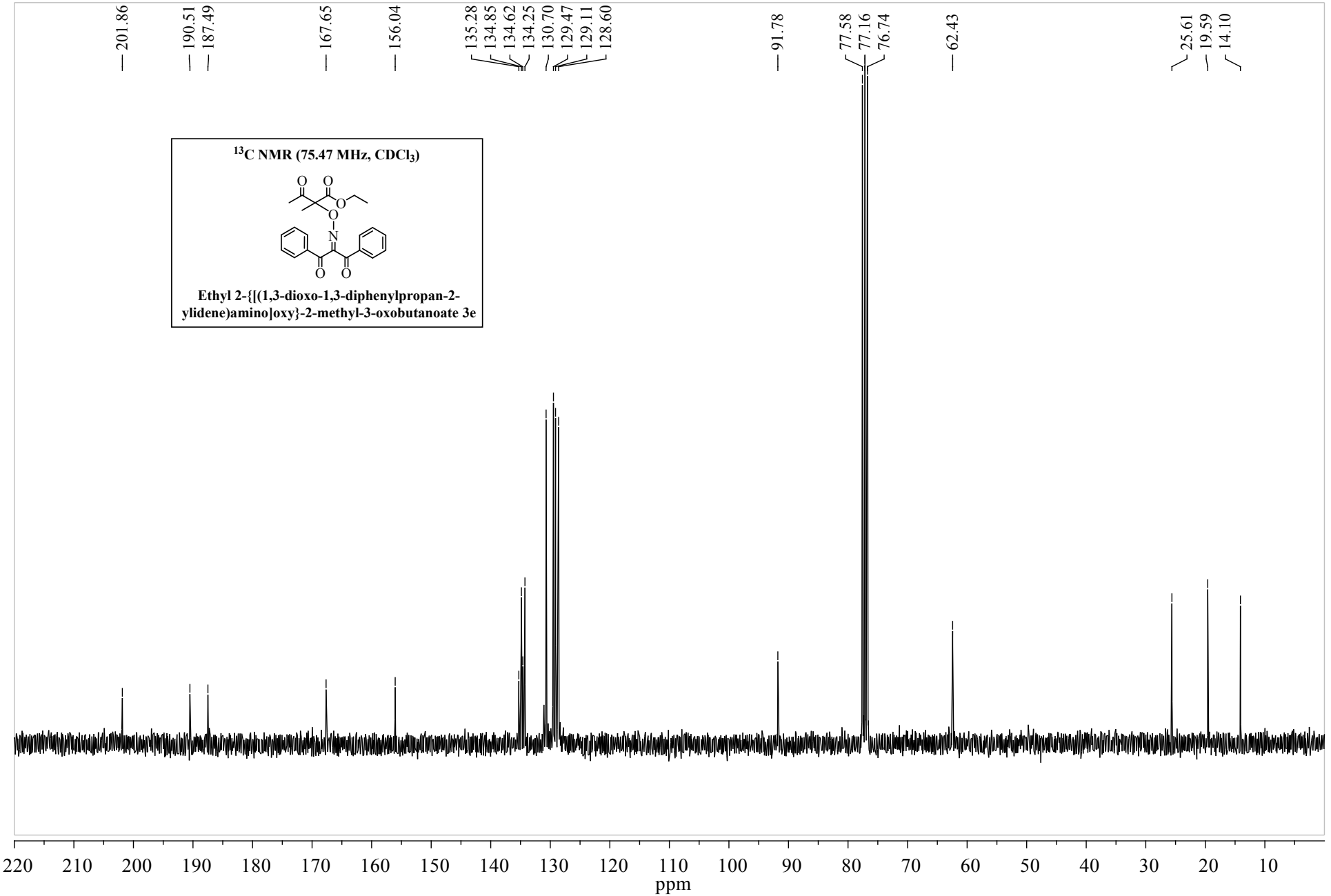

7.26
4.45
4.42
4.40
4.37
4.36
4.34
4.31
4.29
4.28
4.26
4.24
4.23
4.22
4.19
2.24
1.72
1.40
1.37
1.35
1.32
1.30
1.28
1.26
1.24
$^1$H NMR (300.13 MHz, $CDCl_3$)
Diethyl 2-{[(1-ethoxy-2-methyl-1,3-dioxobutan-2-yl)oxy]imino}malonate 3c
6.00
2.94
2.88
9.40
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

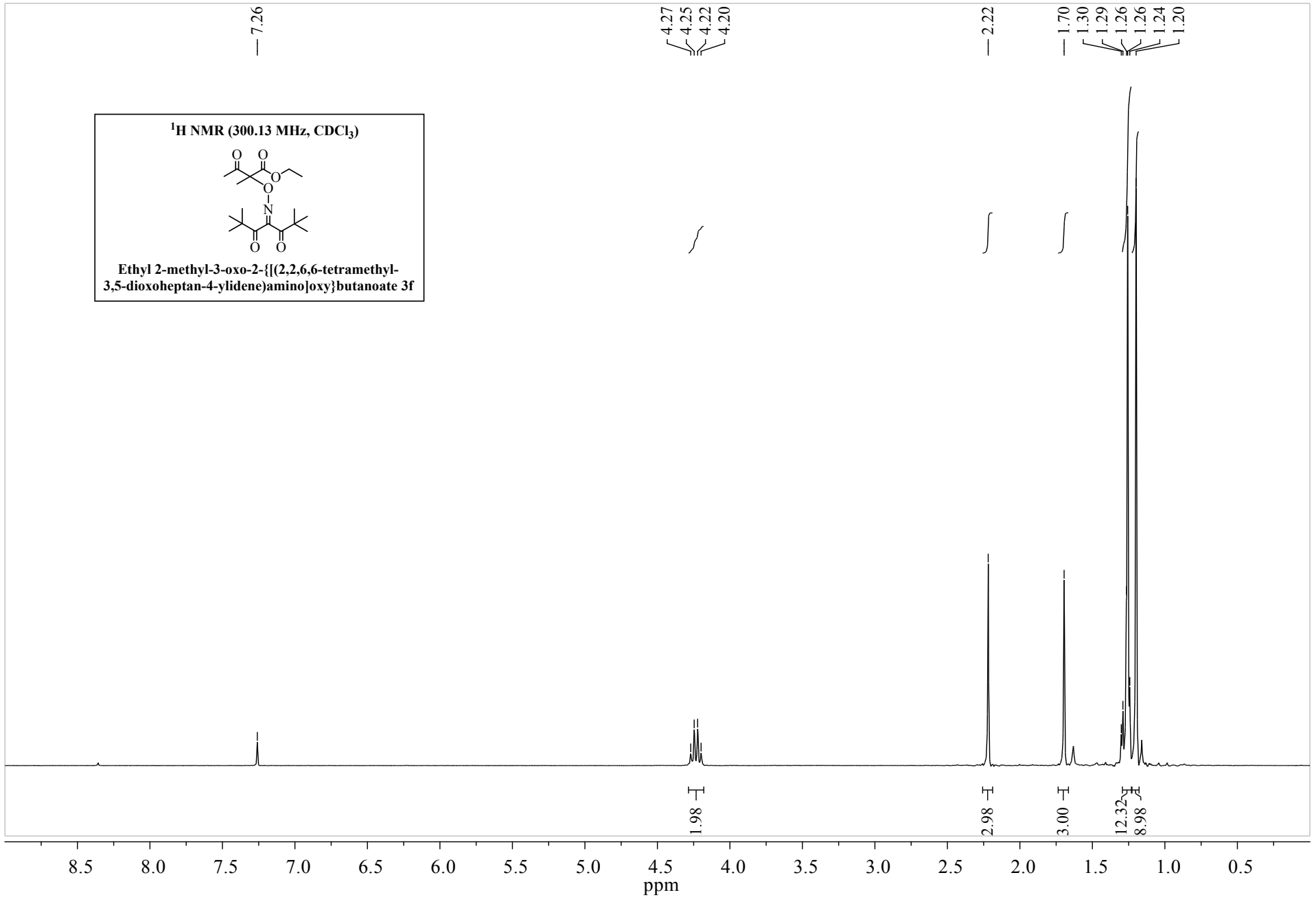

201.78
167.14
160.04
159.22
146.00
91.94
77.58
77.16
76.74
62.82
62.61
62.40
25.78
19.32
14.22
14.07
$^{13}$C NMR (75.47 MHz, CDCl$_3$)
Diethyl 2-{[(1-ethoxy-2-methyl-1,3-dioxobutan-2-yl)oxy]imino}malonate 3c
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

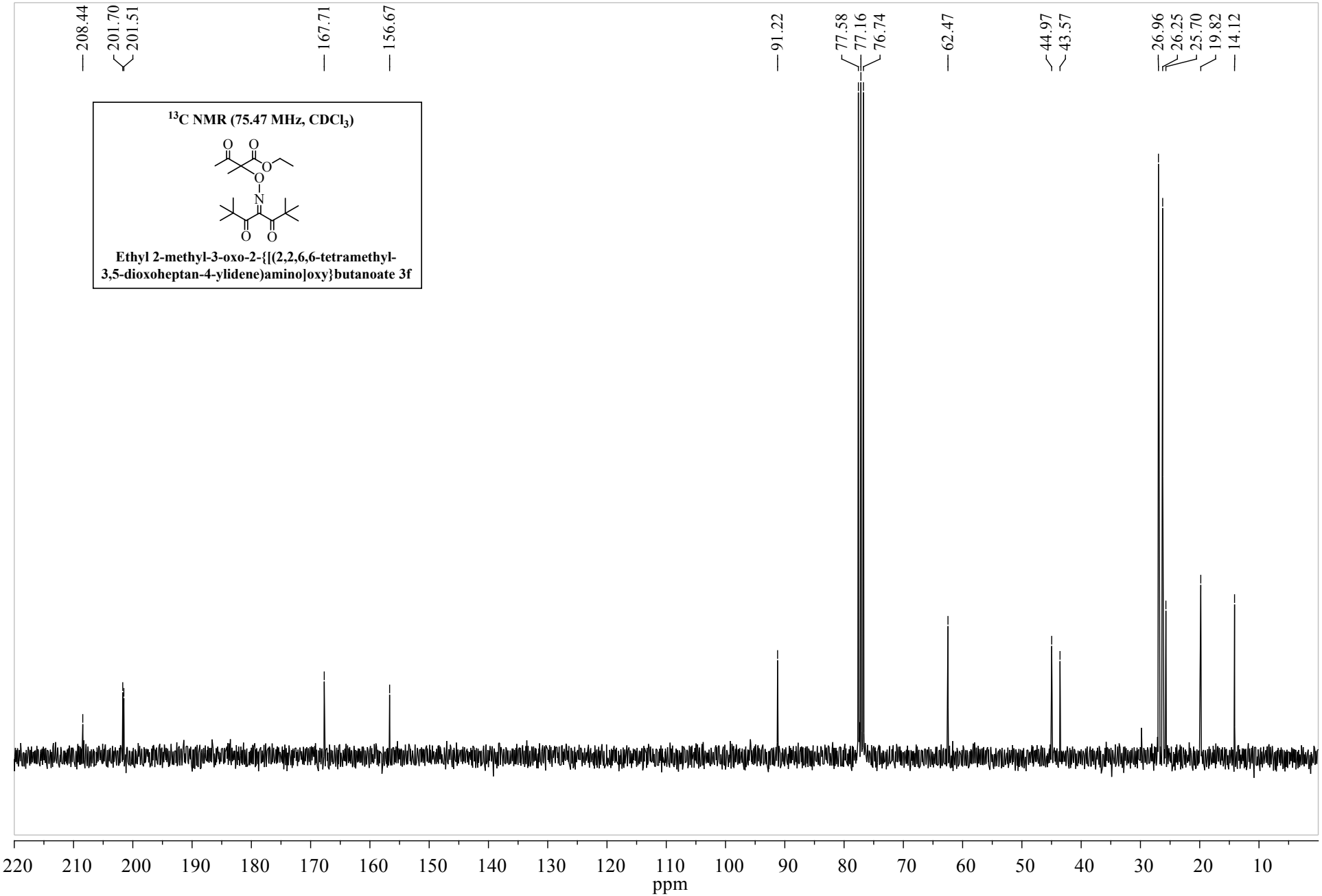

7.26
4.36
4.33
4.31
4.28
4.27
4.26
4.25
4.24
4.23
4.21
4.20
4.19
4.17
4.16
4.15
2.25
2.22
2.15
2.05
1.68
1.61
1.37
1.35
1.31
1.29
1.27
1.27
1.23
$^1$H NMR (300.13 MHz, CDCl$_3$)
Ethyl 2-{[(1-ethoxy-1-oxopropan-2-ylidene)amino]oxy}-2-methyl-3-oxobutanoate (mixture of E and Z isomers 10:1) 3d
4.66
2.73
0.27
2.73
0.27
2.83
0.22
7.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

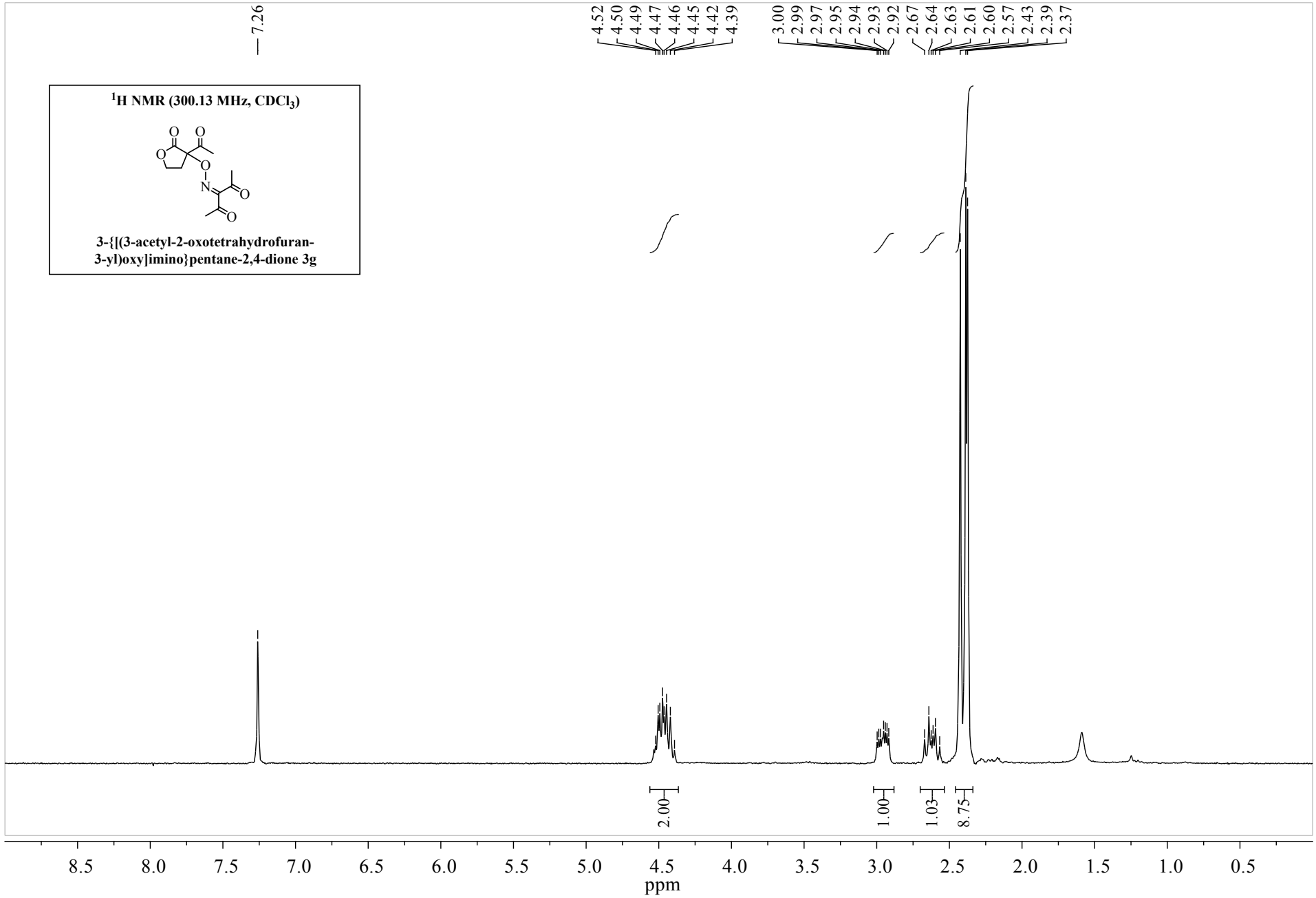

202.79
168.16
163.53
151.42
90.79
77.58
77.16
76.74
62.11
61.89
25.87
19.70
14.17
14.11
11.92
$^{13}$C NMR (75.47 MHz, CDCl$_3$)
Ethyl 2-{[(1-ethoxy-1-oxopropan-2-ylidene)amino]oxy}-2-methyl-3-oxobutanoate (mixture of E and Z isomers 10:1) 3d
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm

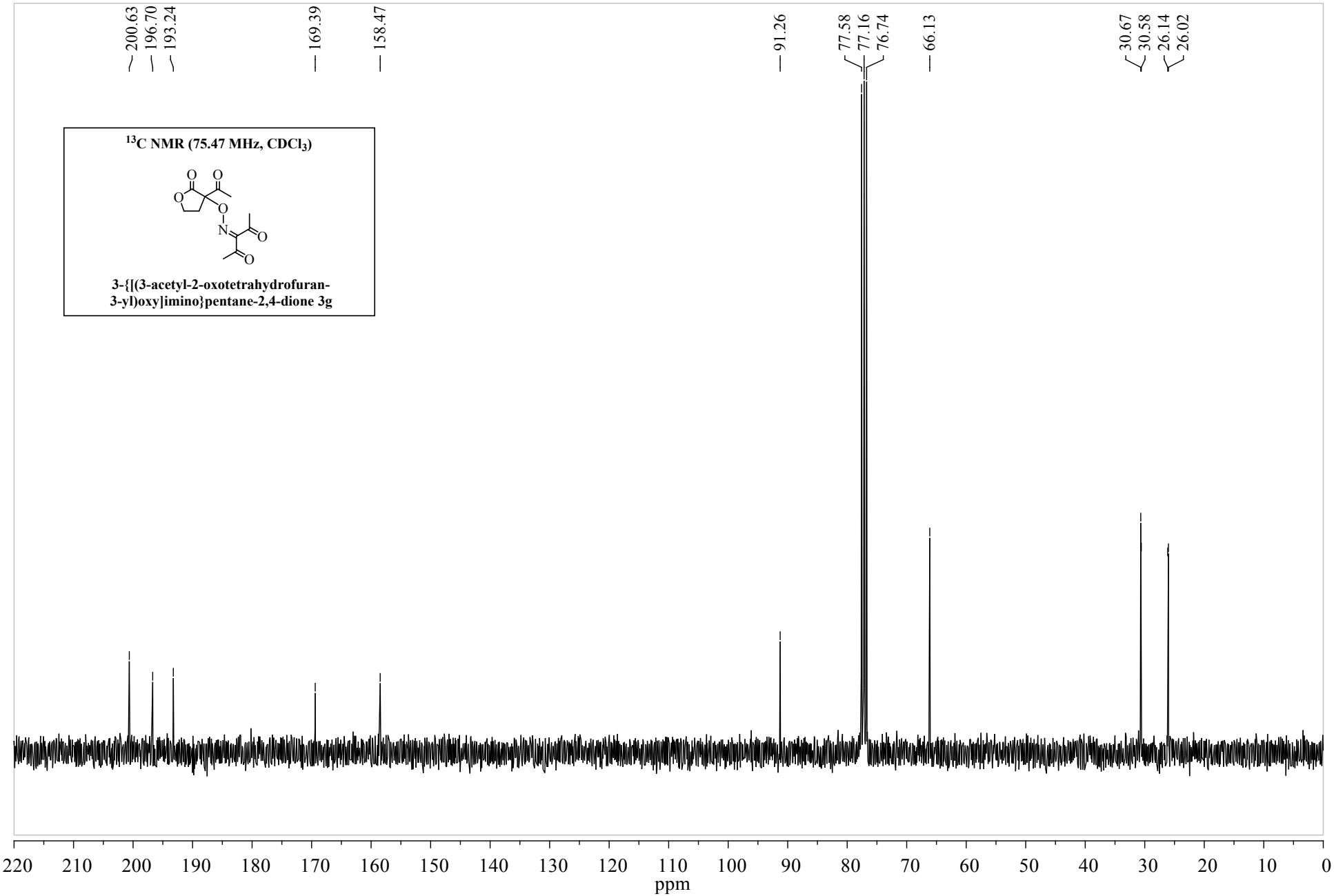

8.11
8.08
8.01
7.98
7.68
7.66
7.63
7.61
7.56
7.53
7.51
7.49
7.46
7.26
4.26
4.23
4.21
4.19
1.96
1.66
1.26
1.24
1.22
1H NMR (300.13 MHz, CDCl3)
Ethyl 2-{[(1,3-dioxo-1,3-diphenylpropan-2-ylidene)amino]oxy}-2-methyl-3-oxobutanoate 3e
1.92
1.88
2.05
4.16
2.00
2.92
3.09
3.03
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

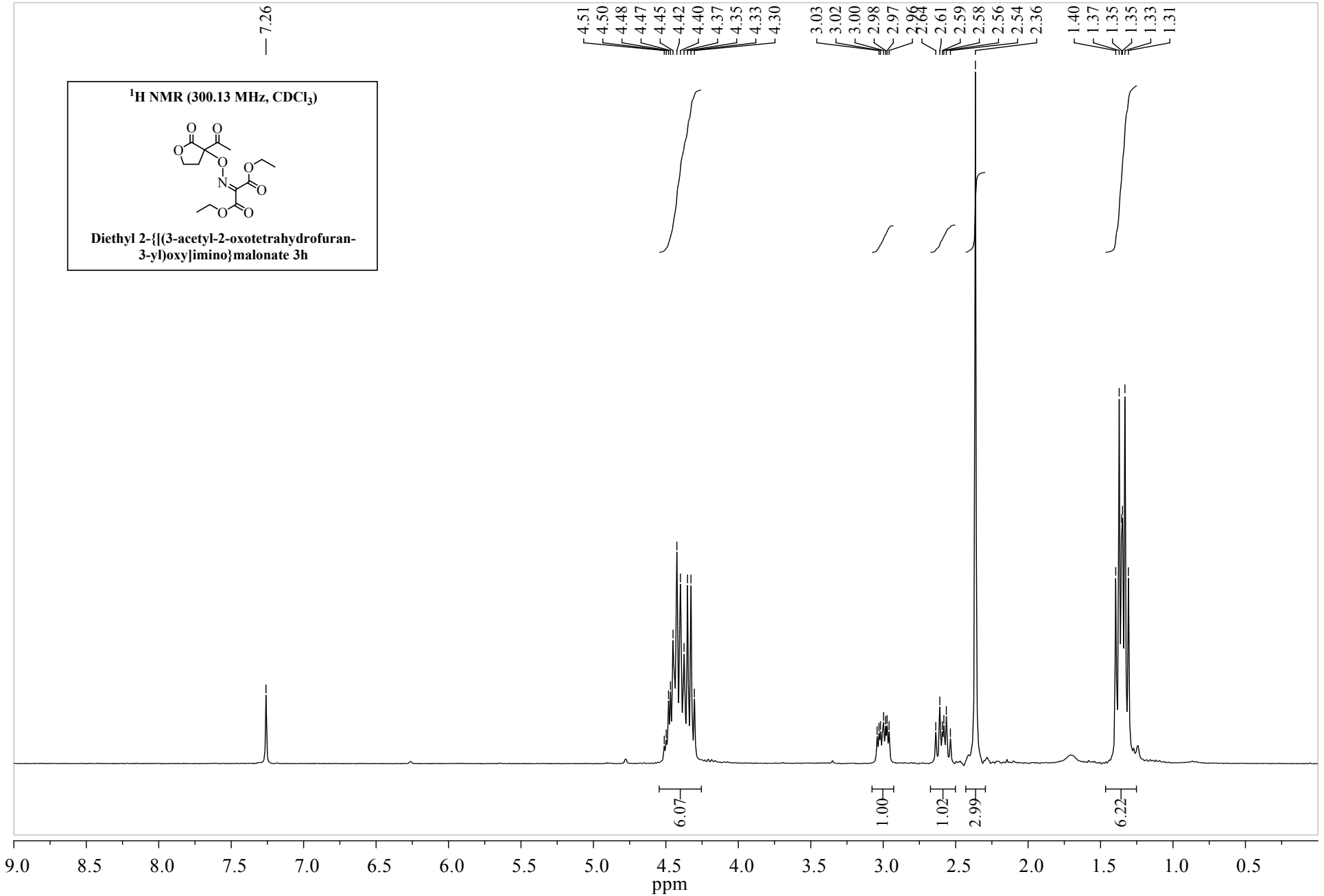

201.86
190.51
187.49
167.65
156.04
135.28
134.85
134.62
134.25
130.70
129.47
129.11
128.60
91.78
77.58
77.16
76.74
62.43
25.61
19.59
14.10
13C NMR (75.47 MHz, CDCl3)
Ethyl 2-{[(1,3-dioxo-1,3-diphenylpropan-2-ylidene)amino]oxy}-2-methyl-3-oxobutanoate 3e
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm


7.26
4.27
4.25
4.22
4.20
2.22
1.70
1.30
1.29
1.26
1.26
1.24
1.20
$^1$H NMR (300.13 MHz, CDCl$_3$)
Ethyl 2-methyl-3-oxo-2-{[(2,2,6,6-tetramethyl-3,5-dioxoheptan-4-ylidene)amino]oxy}butanoate 3f
1.98
2.98
3.00
12.32
8.98
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm


208.44
201.70
201.51
167.71
156.67
91.22
77.58
77.16
76.74
62.47
44.97
43.57
26.96
26.25
25.70
19.82
14.12
13C NMR (75.47 MHz, CDCl3)
Ethyl 2-methyl-3-oxo-2-{[(2,2,6,6-tetramethyl-3,5-dioxoheptan-4-ylidene)amino]oxy}butanoate 3f
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm

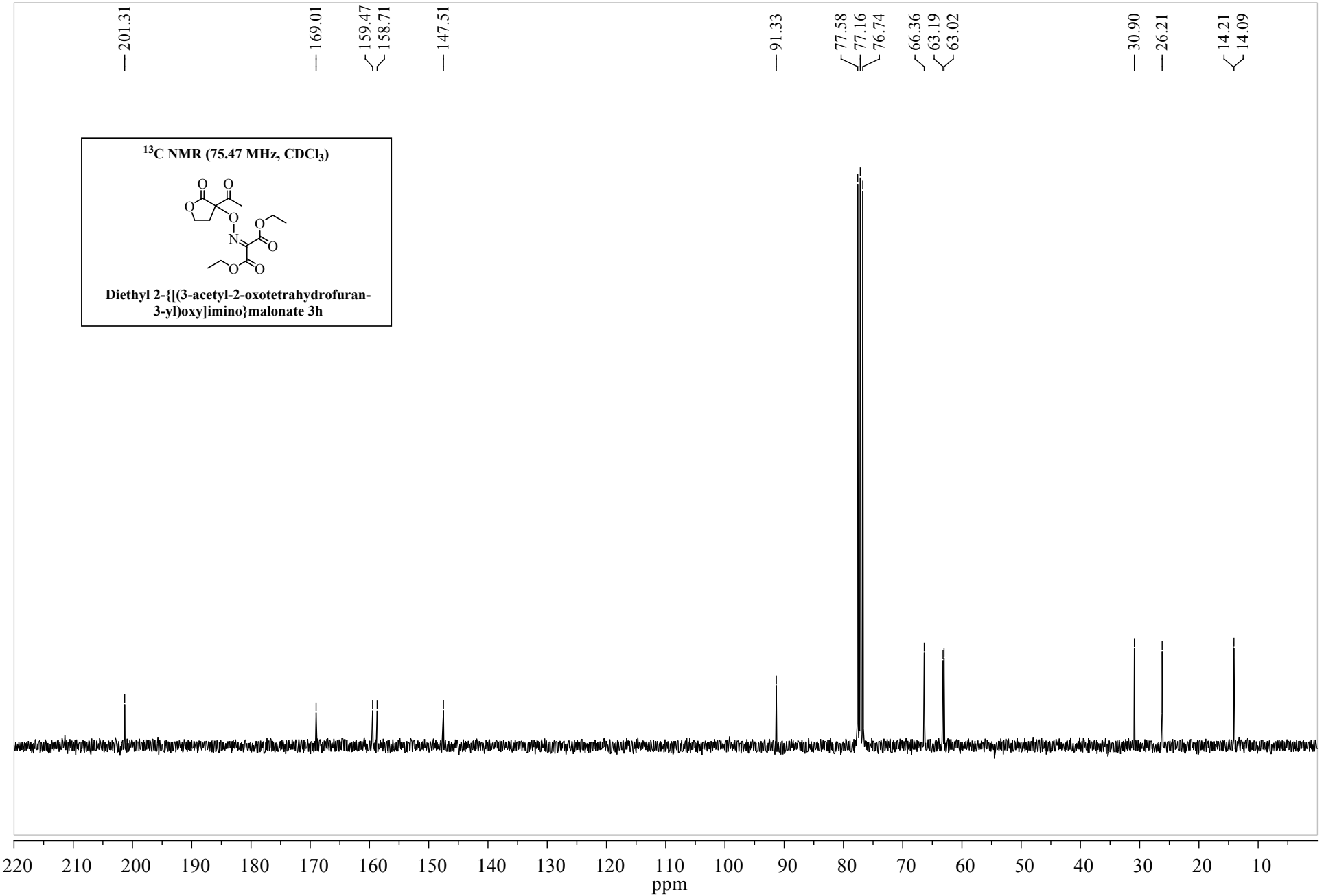

7.26
4.52
4.50
4.49
4.47
4.46
4.45
4.42
4.39
3.00
2.99
2.97
2.95
2.94
2.93
2.92
2.67
2.64
2.63
2.61
2.60
2.57
2.43
2.39
2.37
$^1$H NMR (300.13 MHz, $CDCl_3$)
3-{[(3-acetyl-2-oxotetrahydrofuran-3-yl)oxy]imino}pentane-2,4-dione 3g
2.00
1.00
1.03
8.75
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

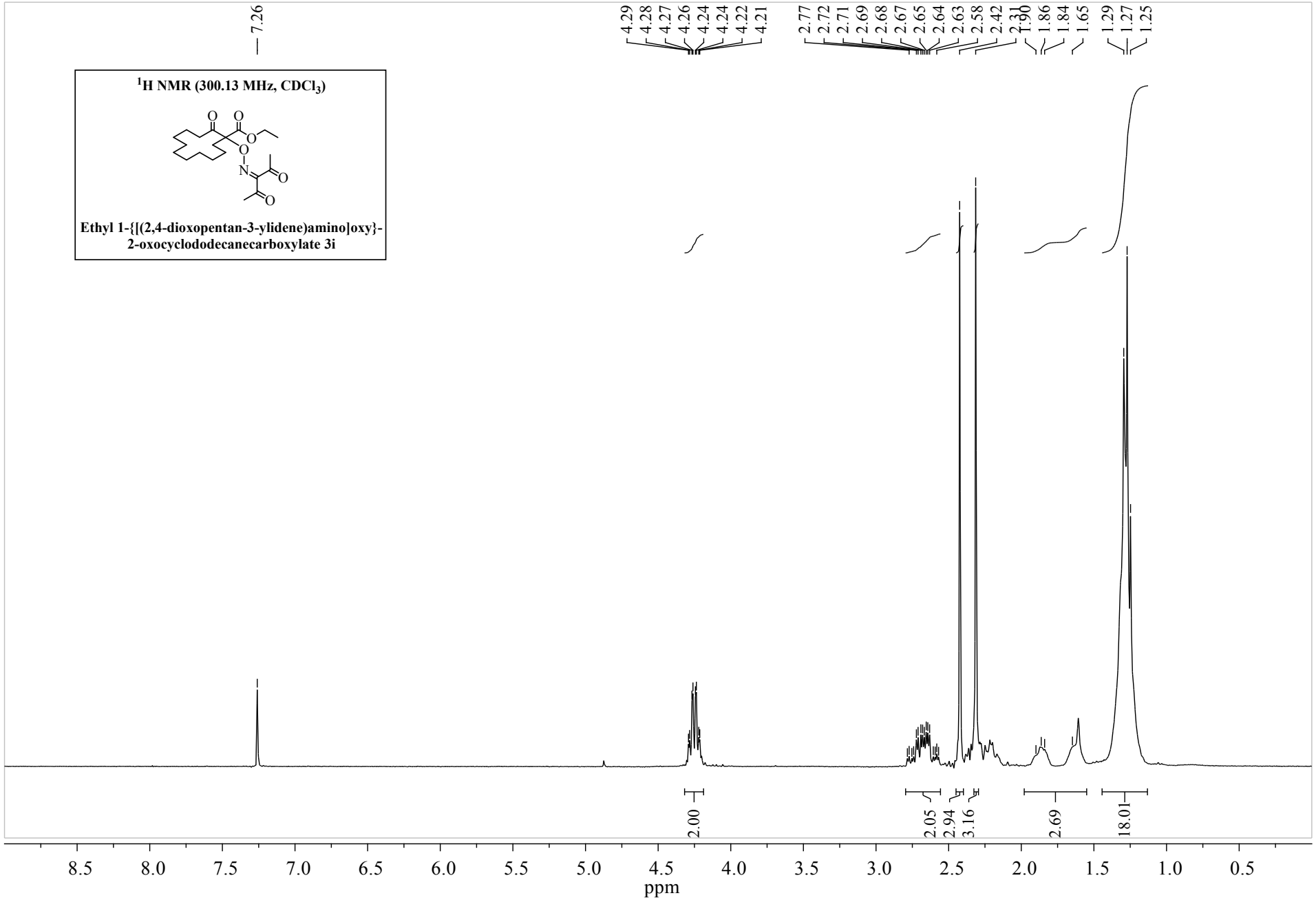

200.63
196.70
193.24
169.39
158.47
91.26
77.58
77.16
76.74
66.13
30.67
30.58
26.14
26.02
$^{13}$C NMR (75.47 MHz, $CDCl_3$)
3-{[(3-acetyl-2-oxotetrahydrofuran-3-yl)oxy]imino}pentane-2,4-dione 3g
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

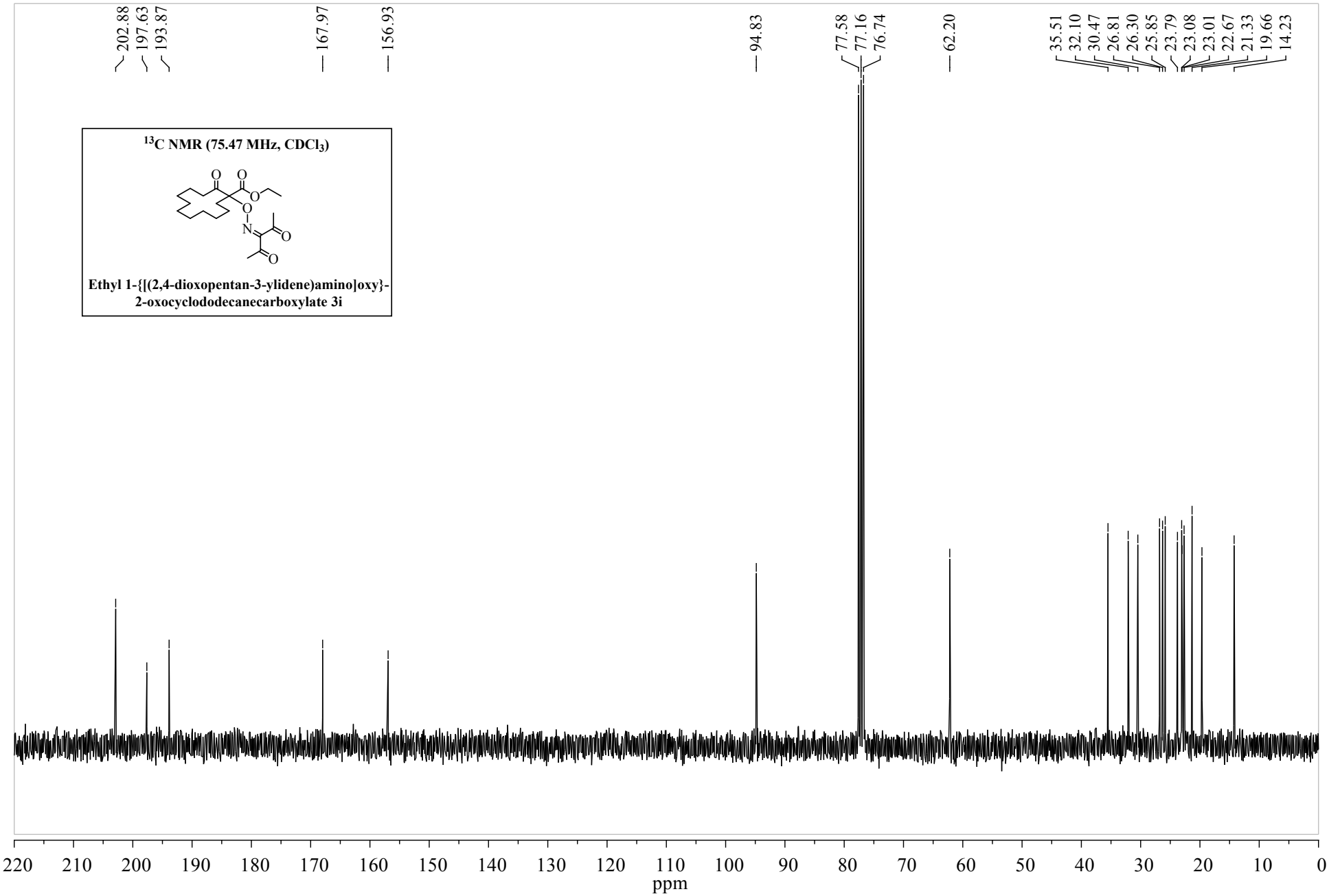



**$^1$H NMR (300.13 MHz, $CDCl_3$)**

**Diethyl 2-{[(3-acetyl-2-oxotetrahydrofuran-3-yl)oxy]imino}malonate 3h**

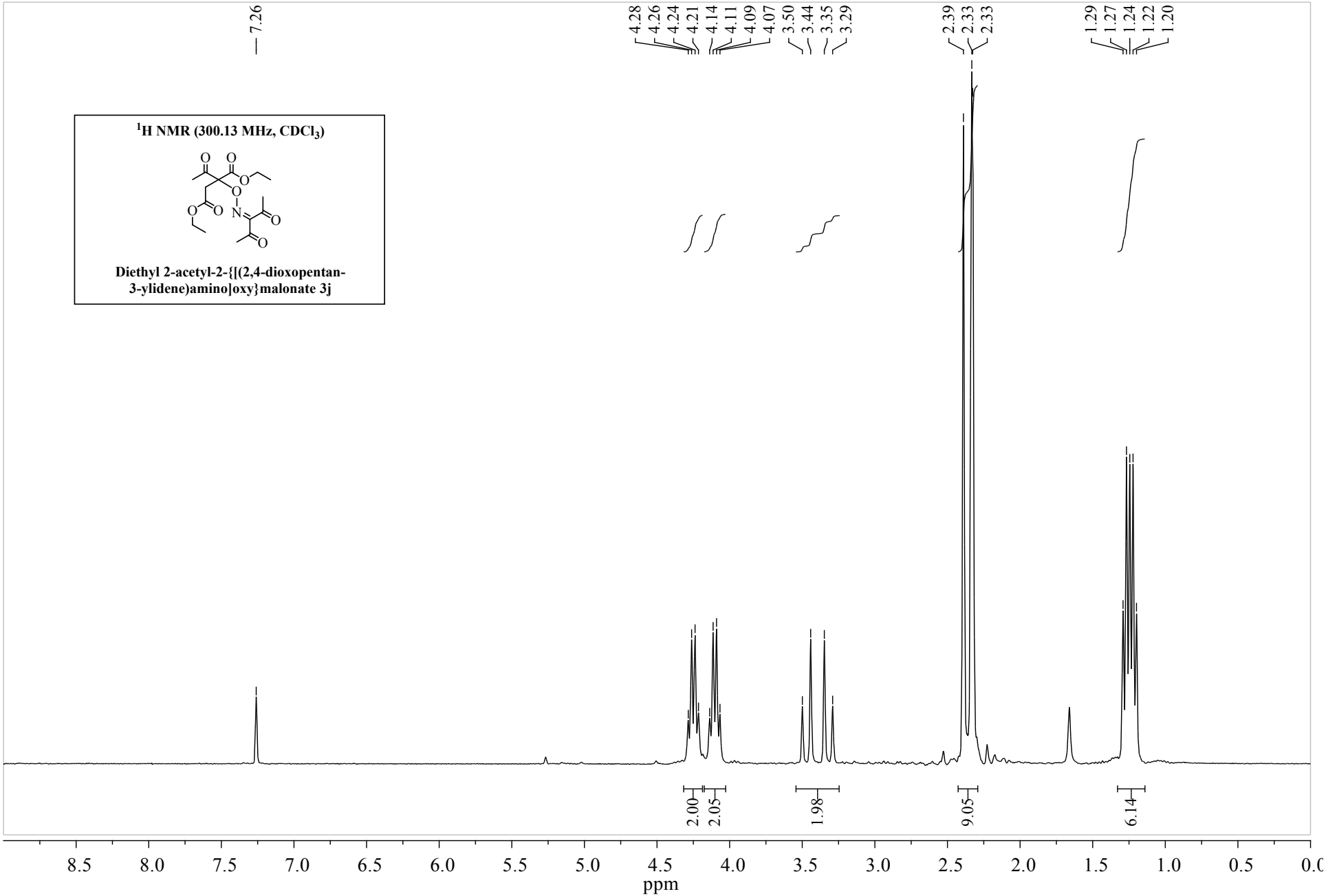

201.31
169.01
159.47
158.71
147.51
91.33
77.58
77.16
76.74
66.36
63.19
63.02
30.90
26.21
14.21
14.09
$^{13}$C NMR (75.47 MHz, $CDCl_3$)
Diethyl 2-{[(3-acetyl-2-oxotetrahydrofuran-3-yl)oxy]imino}malonate 3h
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm


7.26
4.29
4.28
4.27
4.26
4.24
4.24
4.22
4.21
2.77
2.72
2.71
2.69
2.68
2.67
2.65
2.64
2.63
2.58
2.42
2.31
1.90
1.86
1.84
1.65
1.29
1.27
1.25
$^1$H NMR (300.13 MHz, $CDCl_3$)
Ethyl 1-{[(2,4-dioxopentan-3-ylidene)amino]oxy}-2-oxocyclododecanecarboxylate 3i
2.00
2.05
2.94
3.16
2.69
18.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm



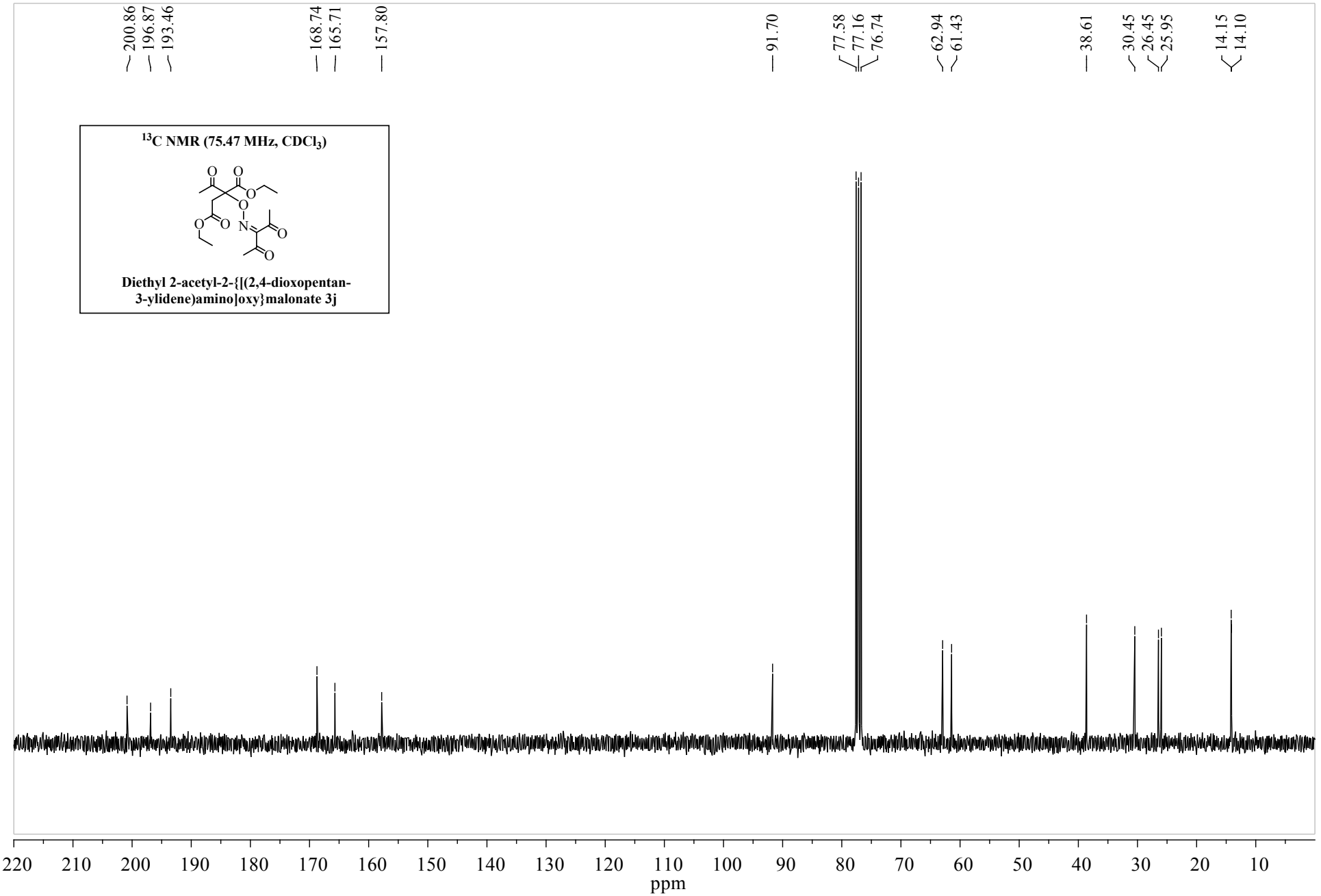

**$^{13}$C NMR (75.47 MHz, $CDCl_3$)**

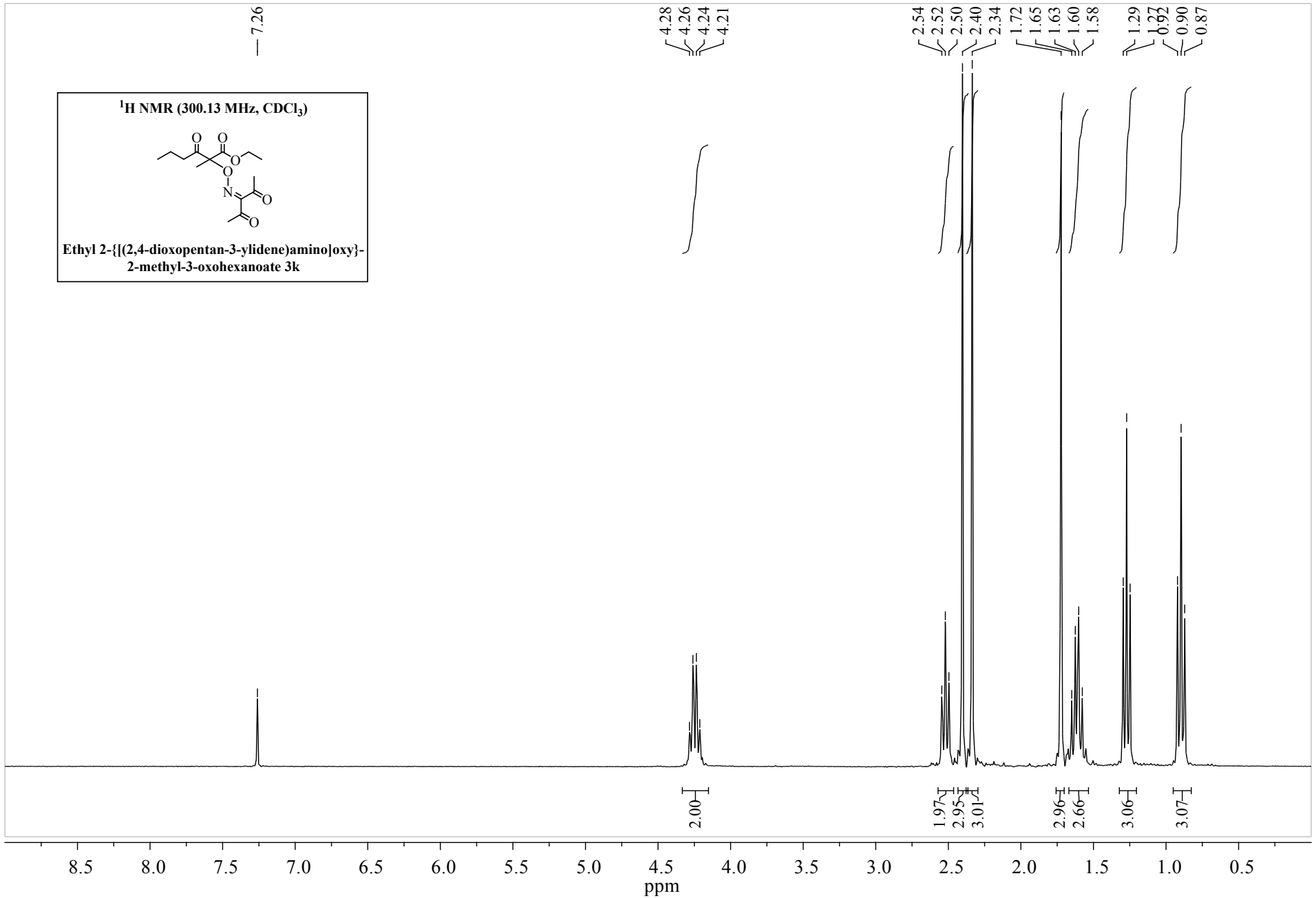
**Ethyl 1-{[(2,4-dioxopentan-3-ylidene)amino]oxy}-2-oxocyclododecanecarboxylate 3i**

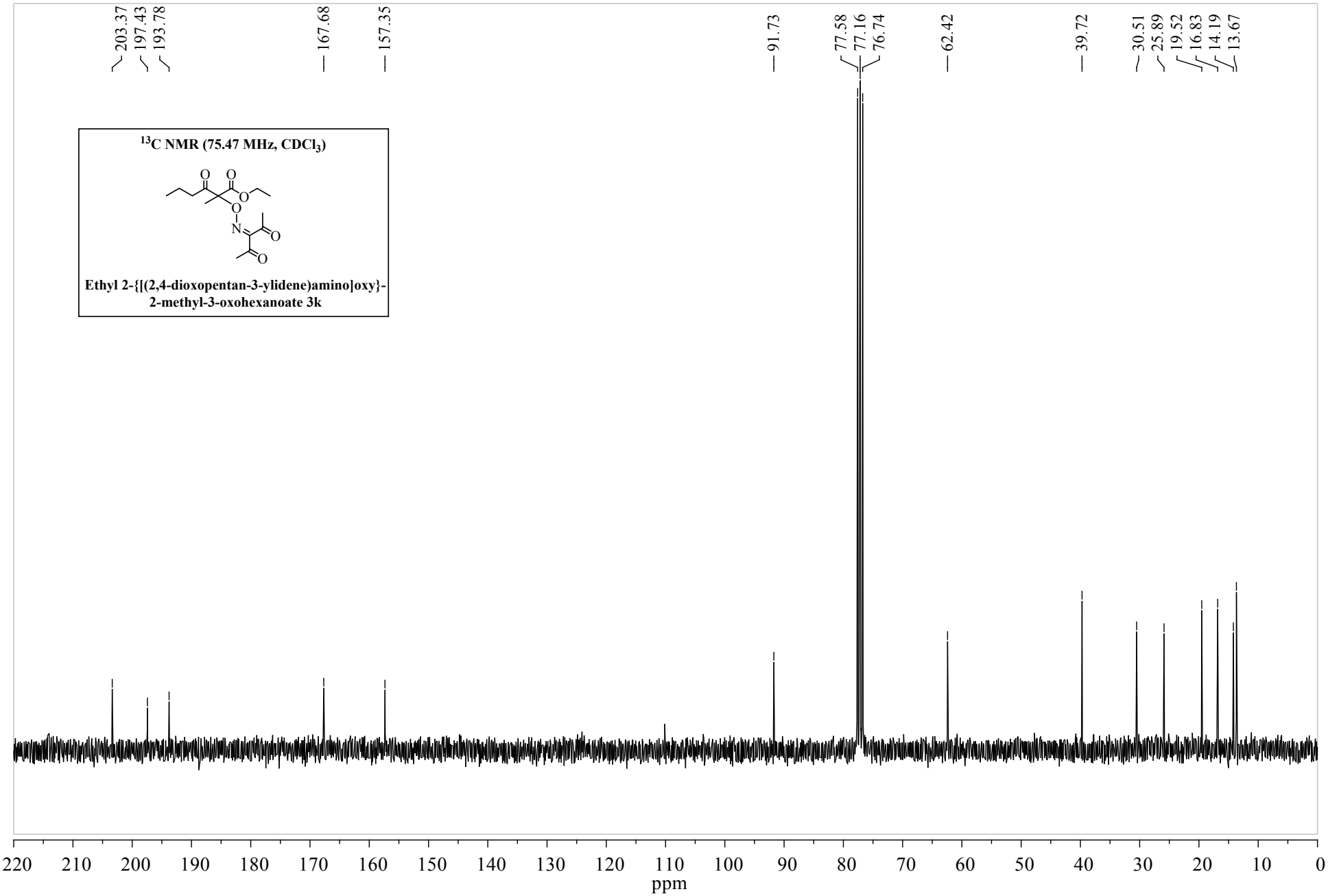


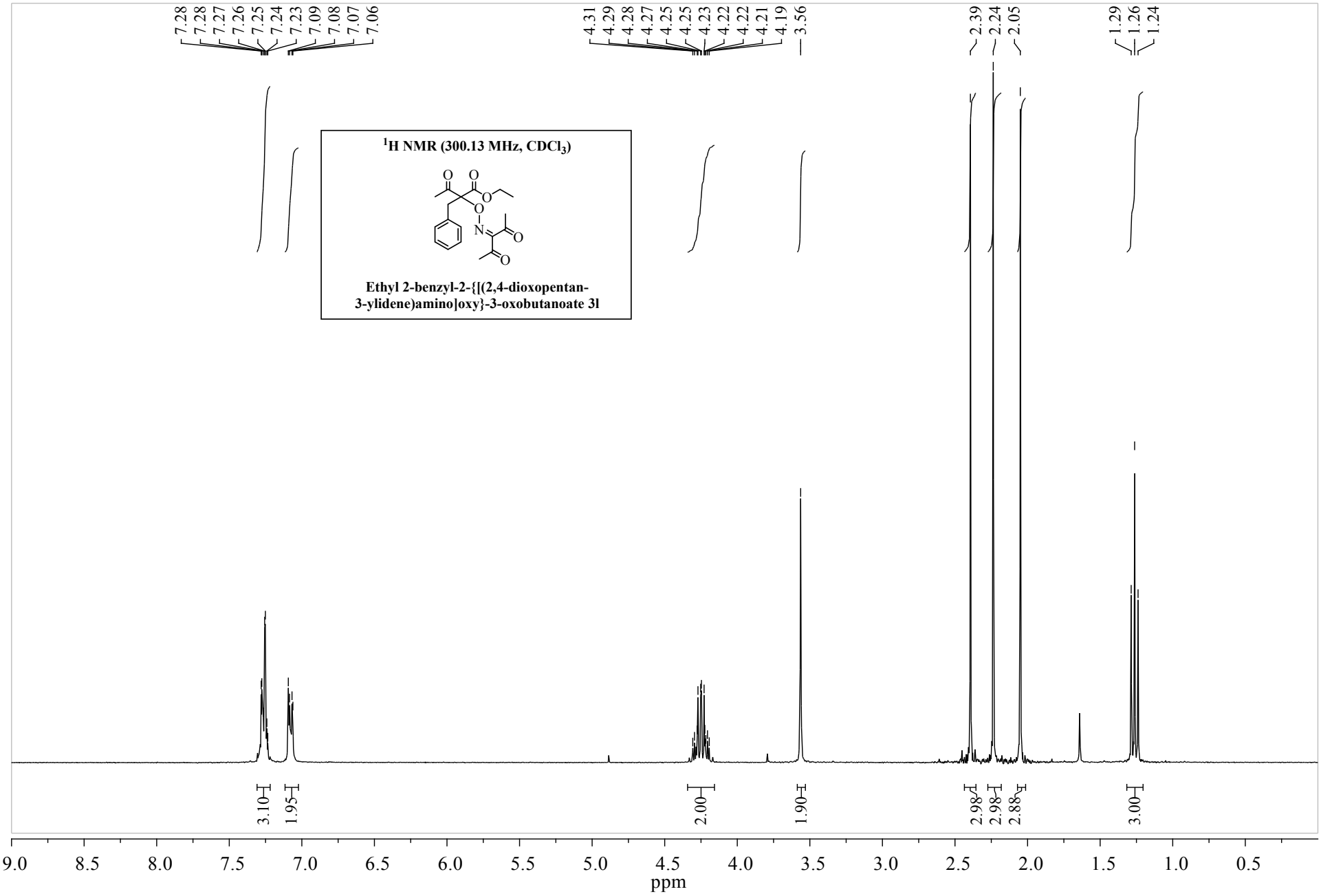

**$^1$H NMR (300.13 MHz, $CDCl_3$)**

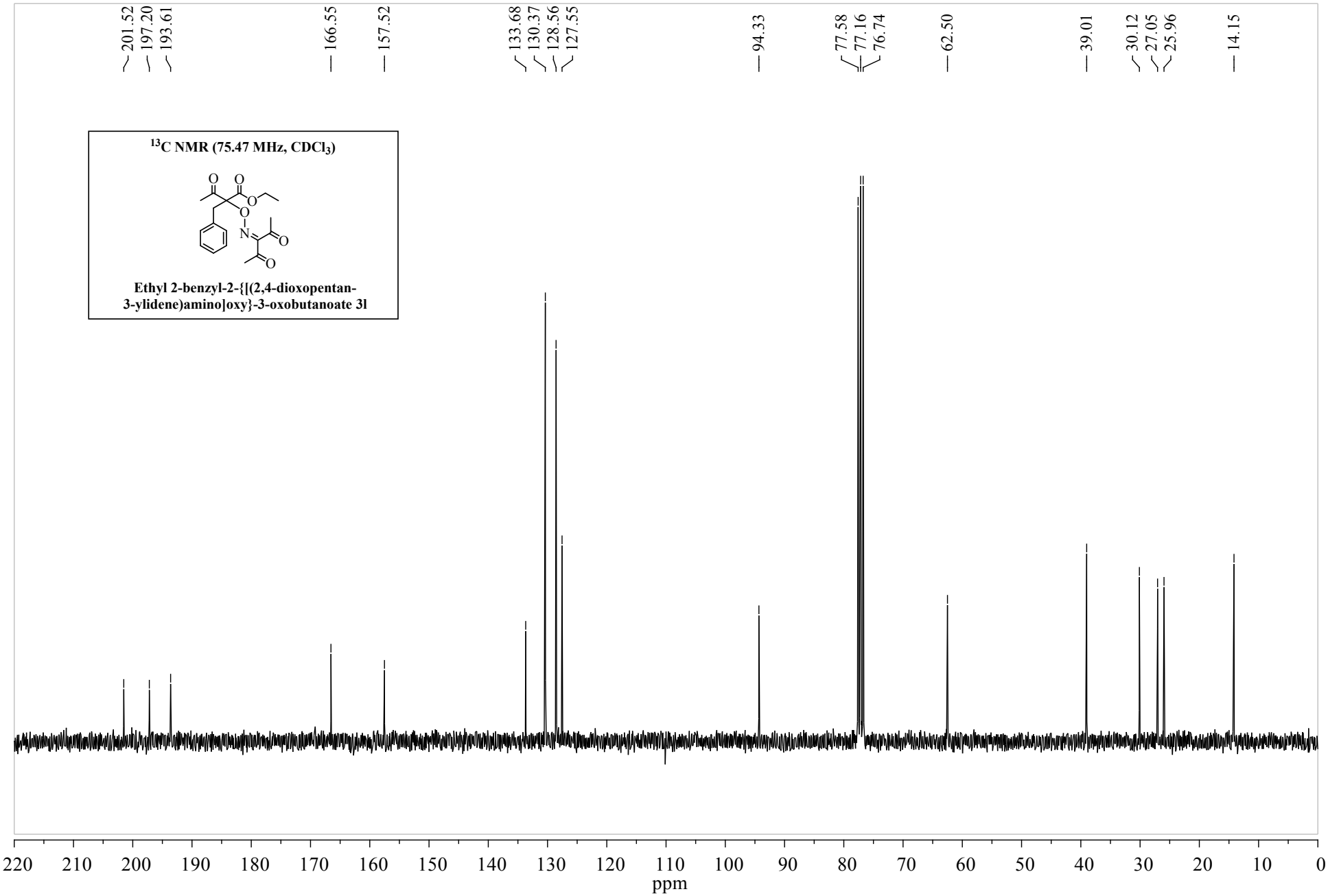
**Diethyl 2-acetyl-2-{[(2,4-dioxopentan-3-ylidene)amino]oxy}malonate 3j**

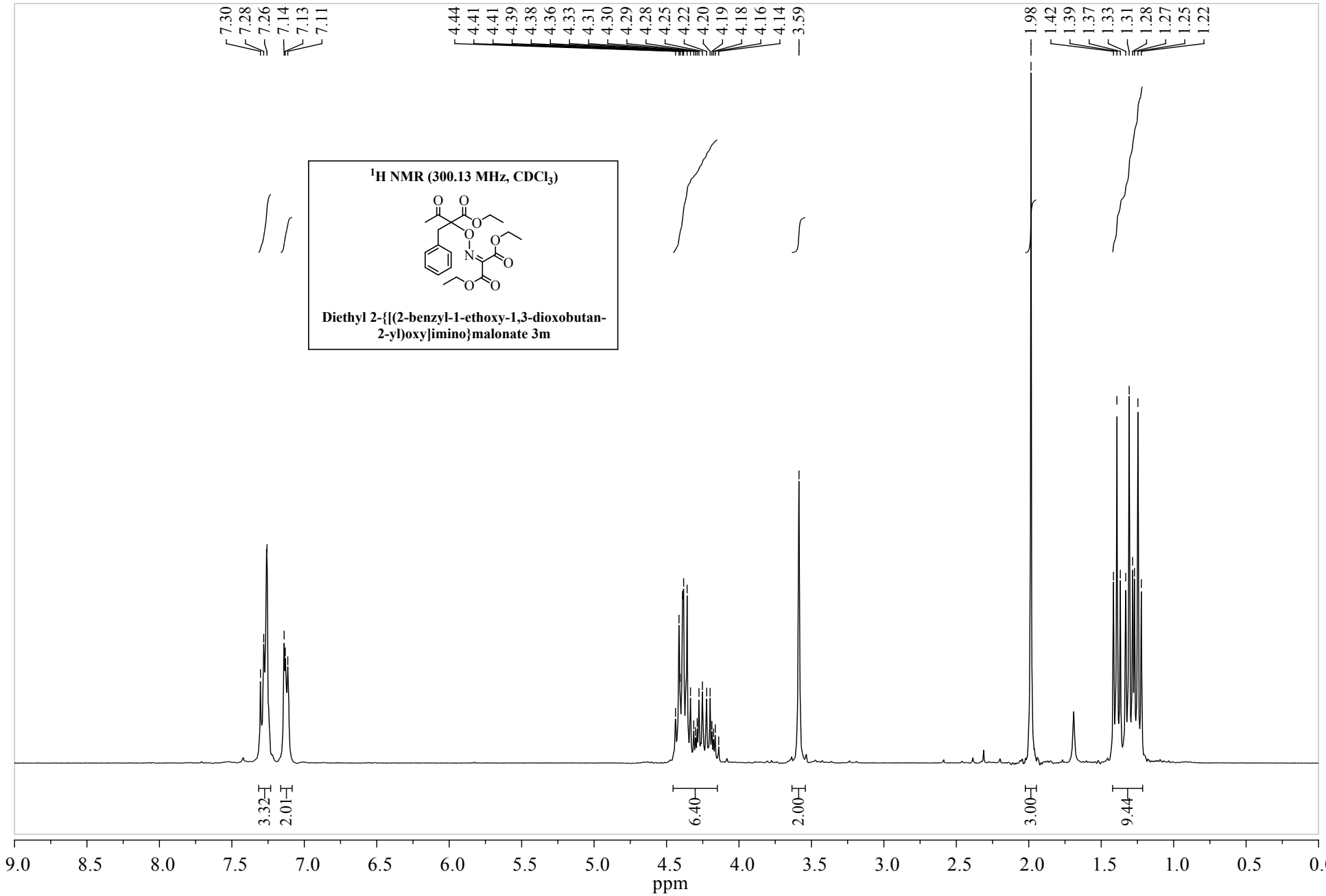

200.86
196.87
193.46
168.74
165.71
157.80
91.70
77.58
77.16
76.74
62.94
61.43
38.61
30.45
26.45
25.95
14.15
14.10
$^{13}$C NMR (75.47 MHz, CDCl$_3$)
Diethyl 2-acetyl-2-{[(2,4-dioxopentan-3-ylidene)amino]oxy}malonate 3j
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm

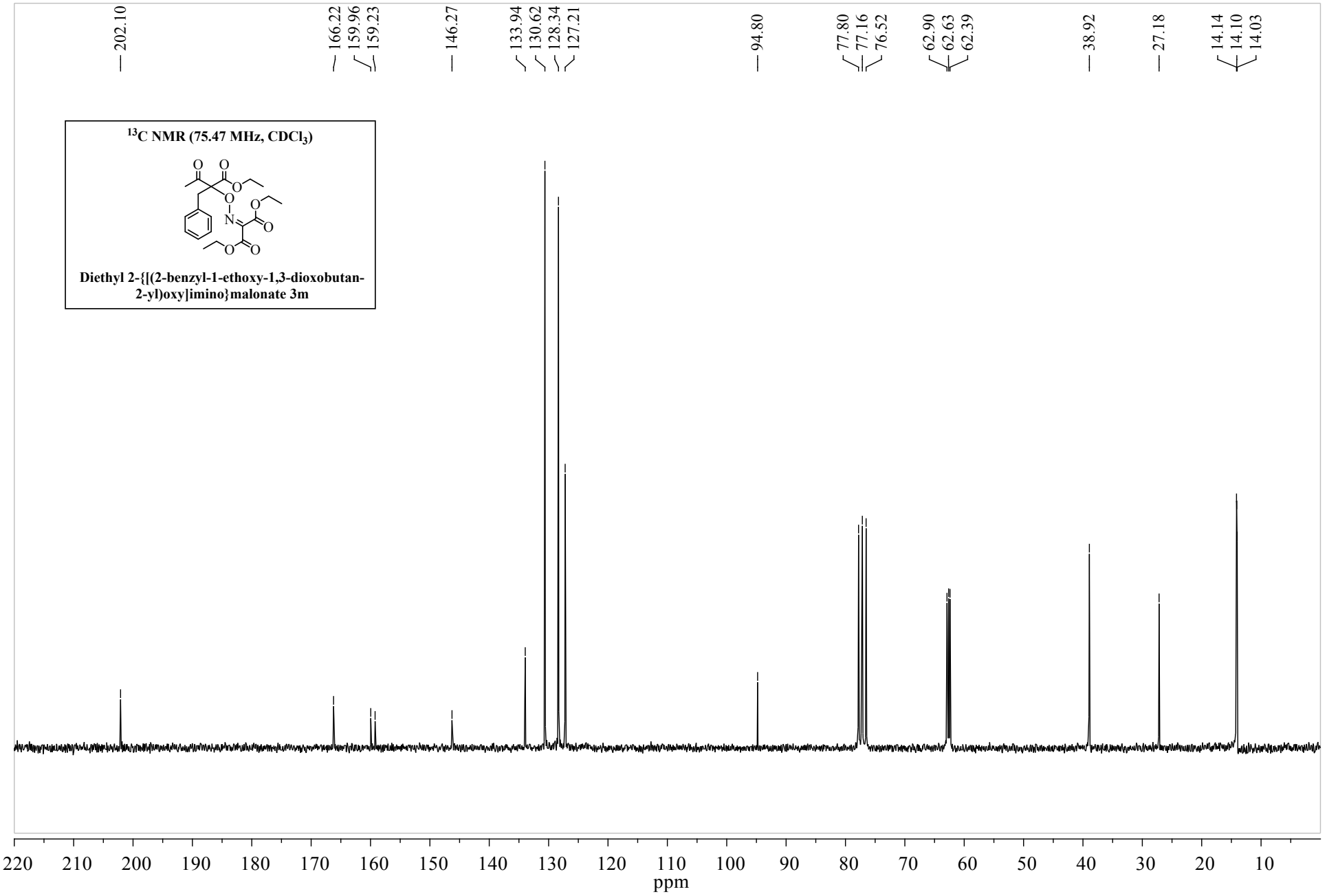

7.26
4.28
4.26
4.24
4.21
2.54
2.52
2.50
2.40
2.34
1.72
1.65
1.63
1.60
1.58
1.29
1.27
0.92
0.90
0.87
$^1$H NMR (300.13 MHz, $CDCl_3$)
Ethyl 2-{[(2,4-dioxopentan-3-ylidene)amino]oxy}-2-methyl-3-oxohexanoate 3k
2.00
1.97
2.95
3.01
2.96
2.66
3.06
3.07
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

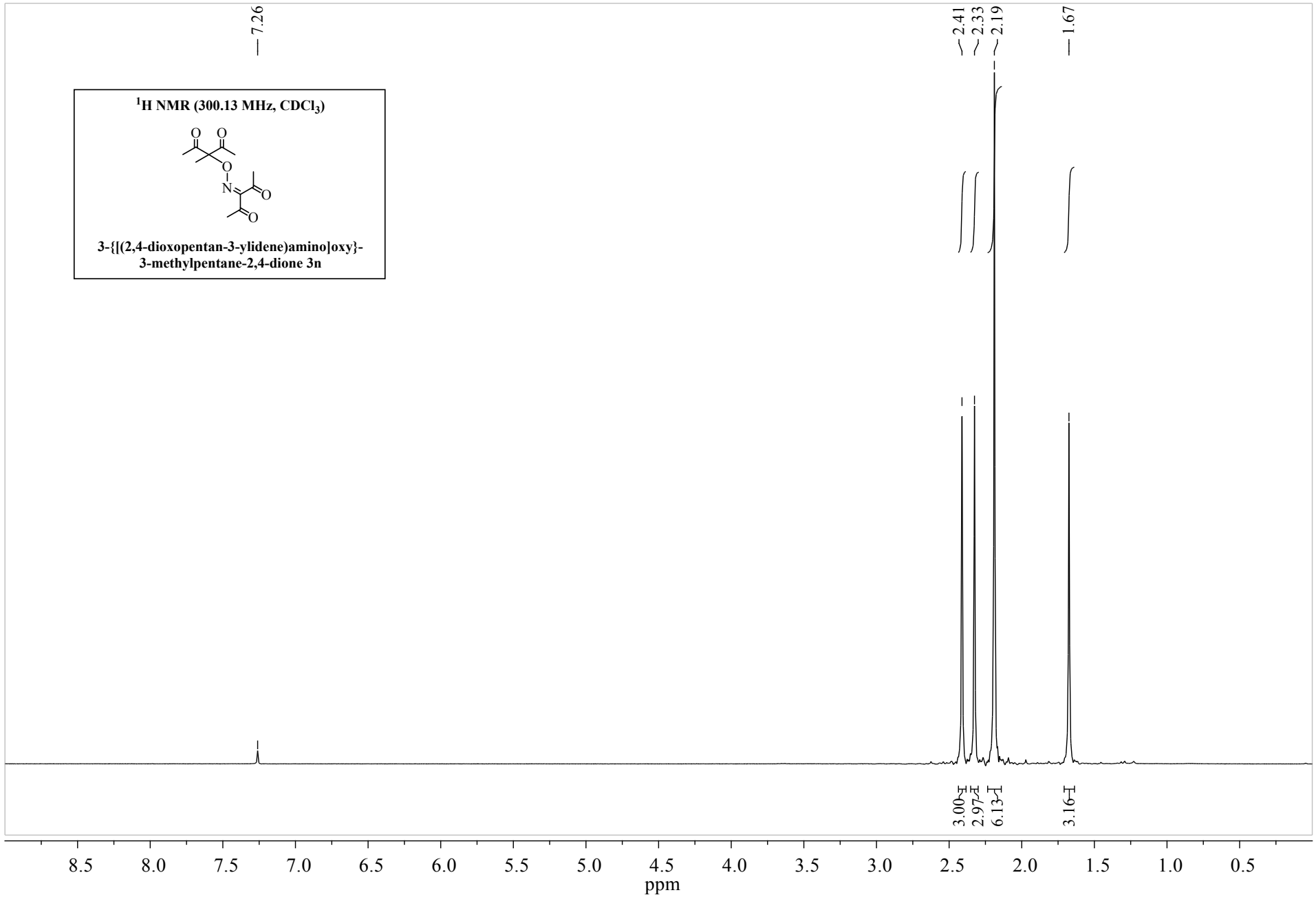

203.37
197.43
193.78
167.68
157.35
91.73
77.58
77.16
76.74
62.42
39.72
30.51
25.89
19.52
16.83
14.19
13.67
$^{13}$C NMR (75.47 MHz, $CDCl_3$)
Ethyl 2-{[(2,4-dioxopentan-3-ylidene)amino]oxy}-2-methyl-3-oxohexanoate 3k
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm


7.28
7.28
7.27
7.26
7.25
7.24
7.23
7.09
7.08
7.07
7.06
4.31
4.29
4.28
4.27
4.25
4.25
4.23
4.22
4.22
4.21
4.19
3.56
2.39
2.24
2.05
1.29
1.26
1.24
$^1$H NMR (300.13 MHz, $CDCl_3$)
Ethyl 2-benzyl-2-{[(2,4-dioxopentan-3-ylidene)amino]oxy}-3-oxobutanoate 3l
3.10
1.95
2.00
1.90
2.98
2.98
2.88
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm


201.52
197.20
193.61
166.55
157.52
133.68
130.37
128.56
127.55
94.33
77.58
77.16
76.74
62.50
39.01
30.12
27.05
25.96
14.15
13C NMR (75.47 MHz, CDCl3)
Ethyl 2-benzyl-2-{[(2,4-dioxopentan-3-ylidene)amino]oxy}-3-oxobutanoate 3l
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

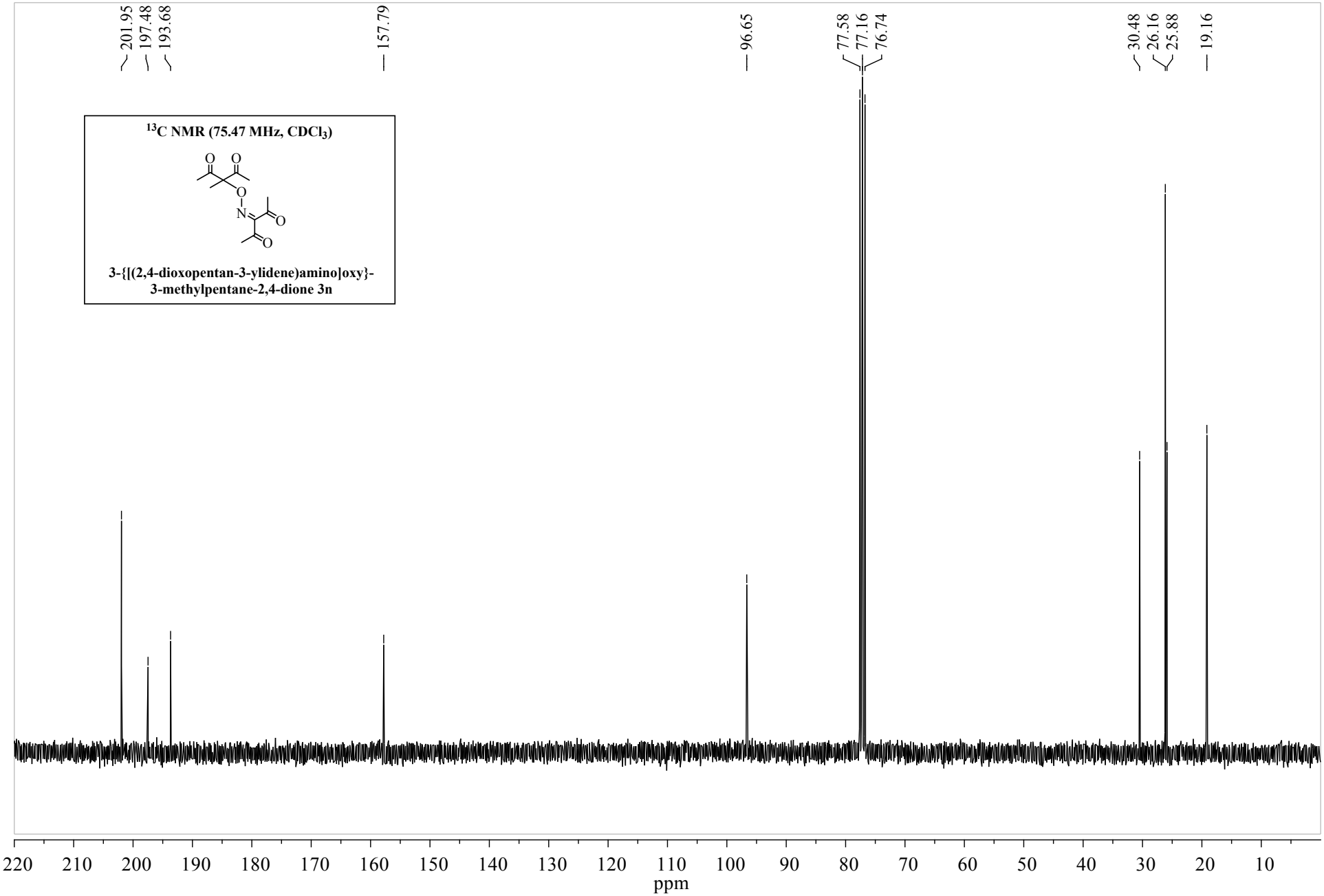

7.30
7.28
7.26
7.14
7.13
7.11
4.44
4.41
4.41
4.39
4.38
4.36
4.33
4.31
4.30
4.29
4.28
4.25
4.22
4.20
4.19
4.18
4.16
4.14
3.59
1.98
1.42
1.39
1.37
1.33
1.31
1.28
1.27
1.25
1.22
$^1$H NMR (300.13 MHz, $CDCl_3$)
Diethyl 2-{[(2-benzyl-1-ethoxy-1,3-dioxobutan-2-yl)oxy]imino}malonate 3m
3.32
2.01
6.40
2.00
3.00
9.44
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

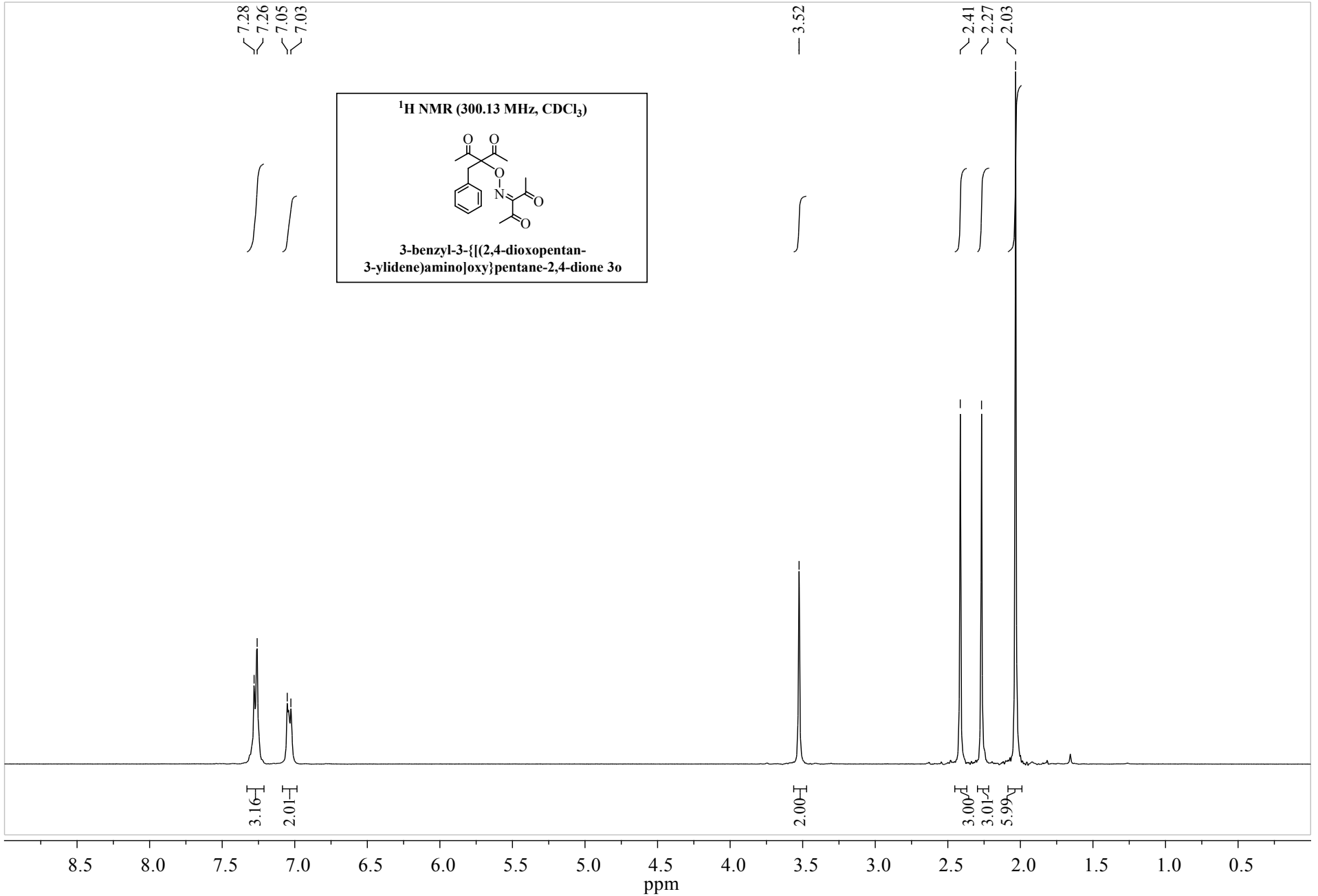

202.10
166.22
159.96
159.23
146.27
133.94
130.62
128.34
127.21
94.80
77.80
77.16
76.52
62.90
62.63
62.39
38.92
27.18
14.14
14.10
14.03
$^{13}$C NMR (75.47 MHz, CDCl$_3$)
Diethyl 2-{[(2-benzyl-1-ethoxy-1,3-dioxobutan-2-yl)oxy]imino}malonate 3m
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm

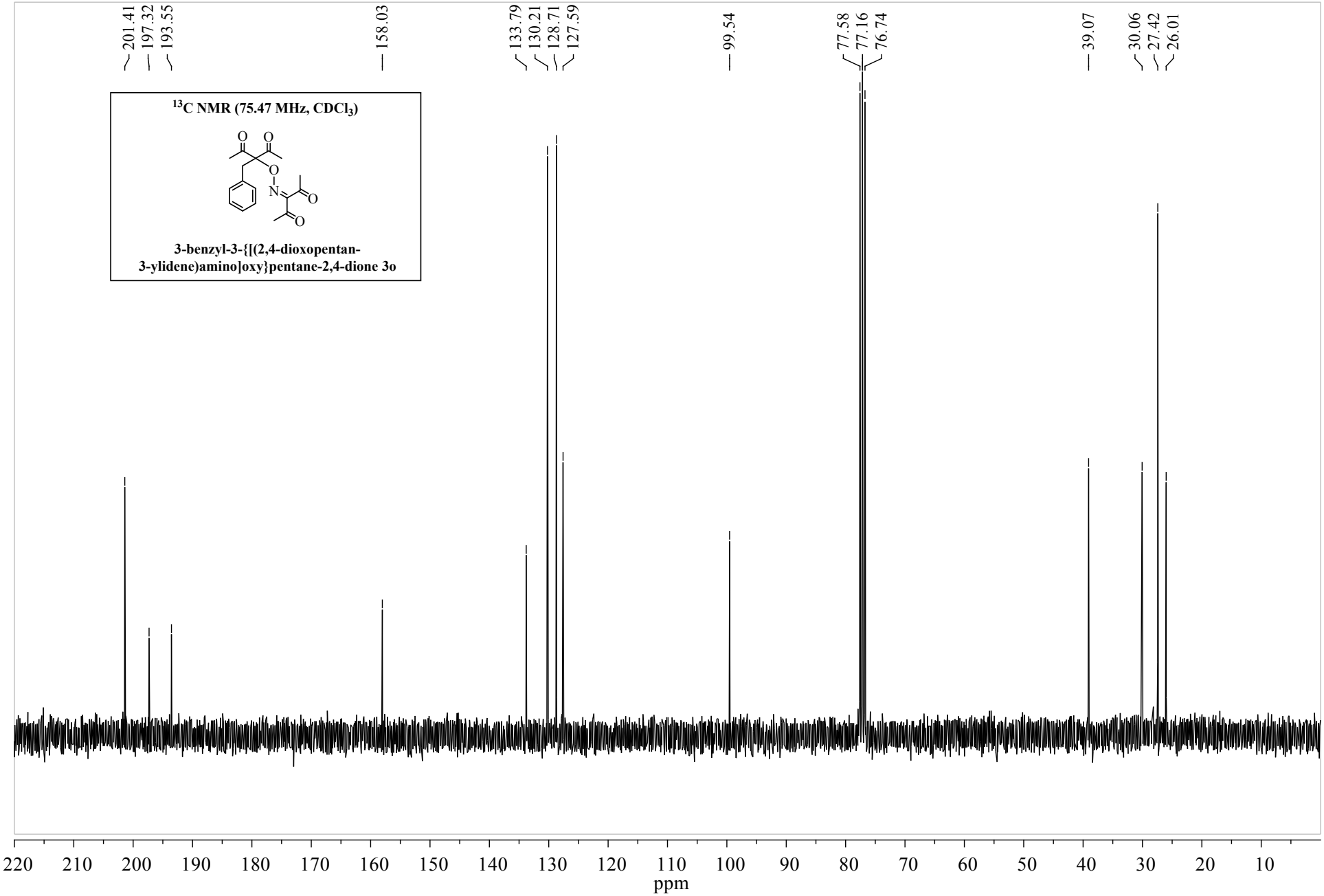


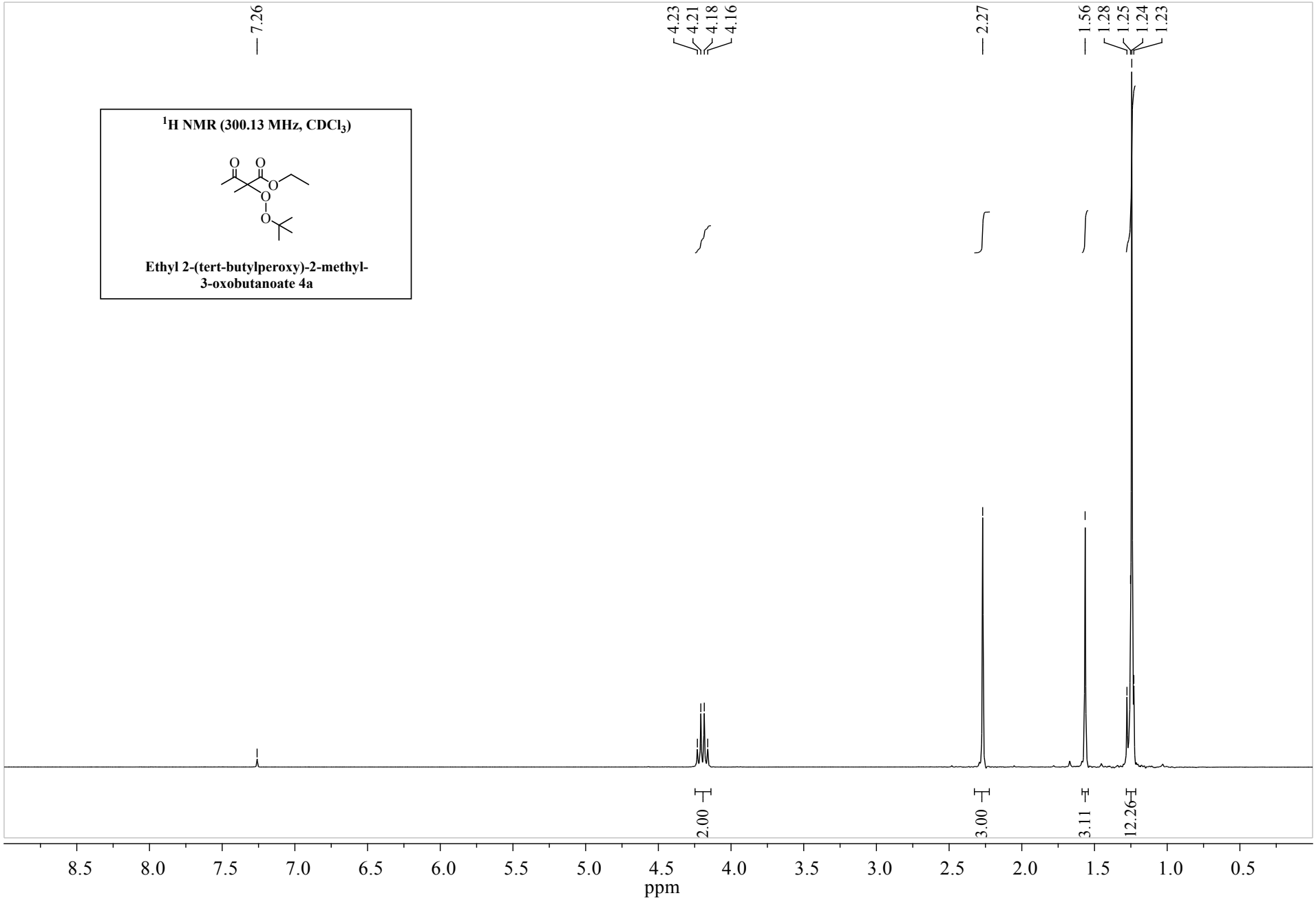

**$^1$H NMR (300.13 MHz, $CDCl_3$)**

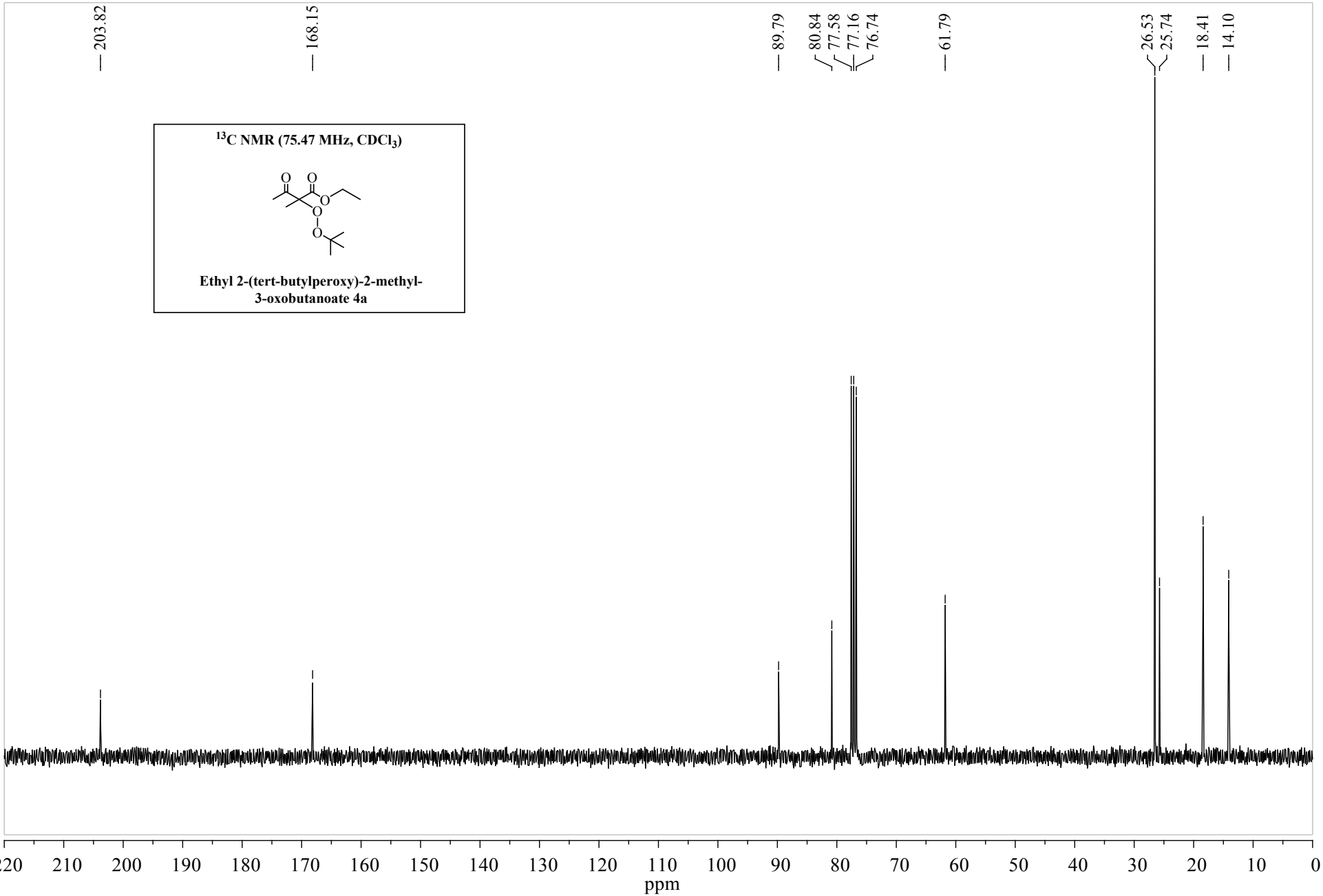
**3-{[(2,4-dioxopentan-3-ylidene)amino]oxy}-3-methylpentane-2,4-dione 3n**

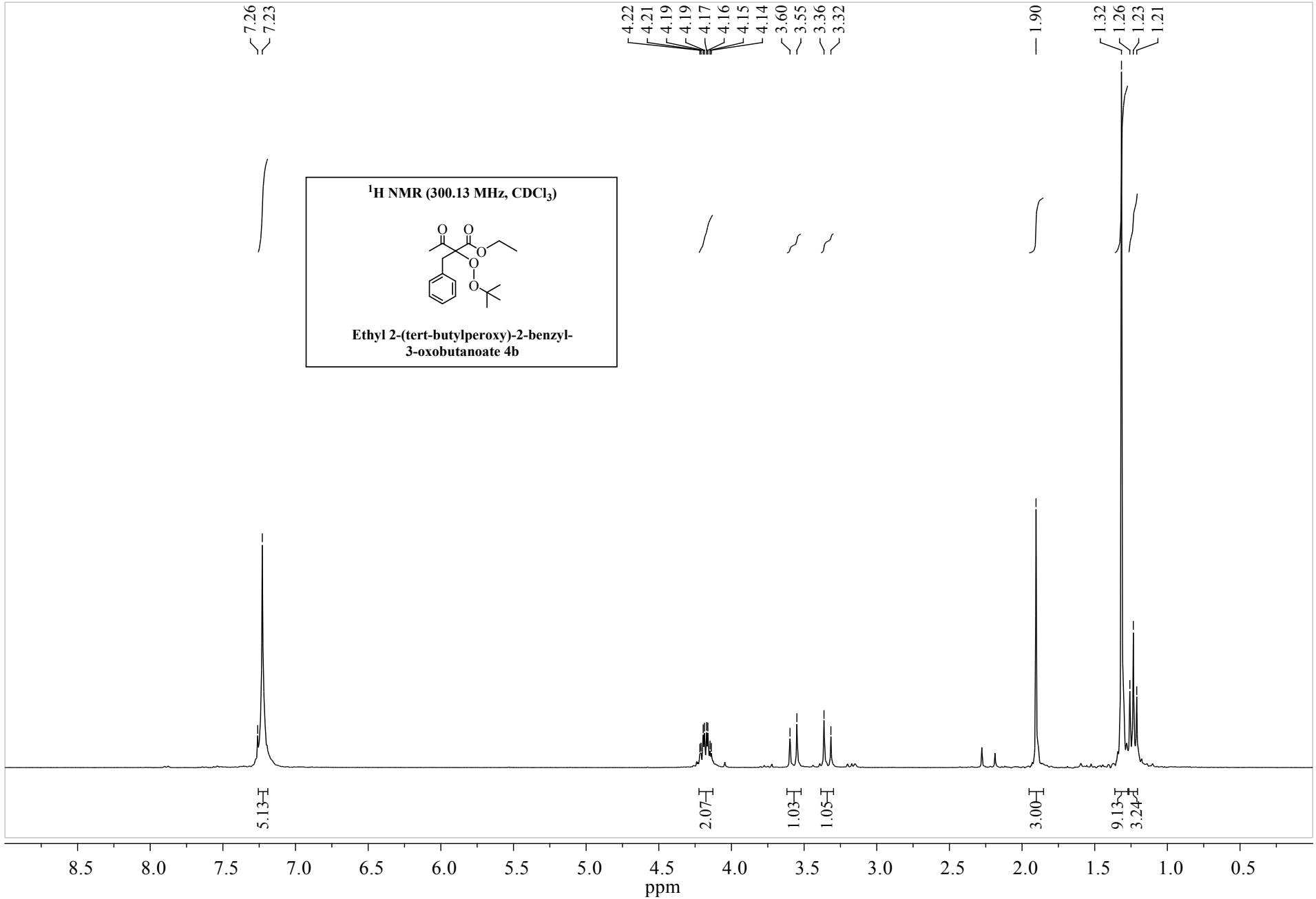

201.95
197.48
193.68
157.79
96.65
77.58
77.16
76.74
30.48
26.16
25.88
19.16
13C NMR (75.47 MHz, CDCl3)
3-{[(2,4-dioxopentan-3-ylidene)amino]oxy}-
3-methylpentane-2,4-dione 3n
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm

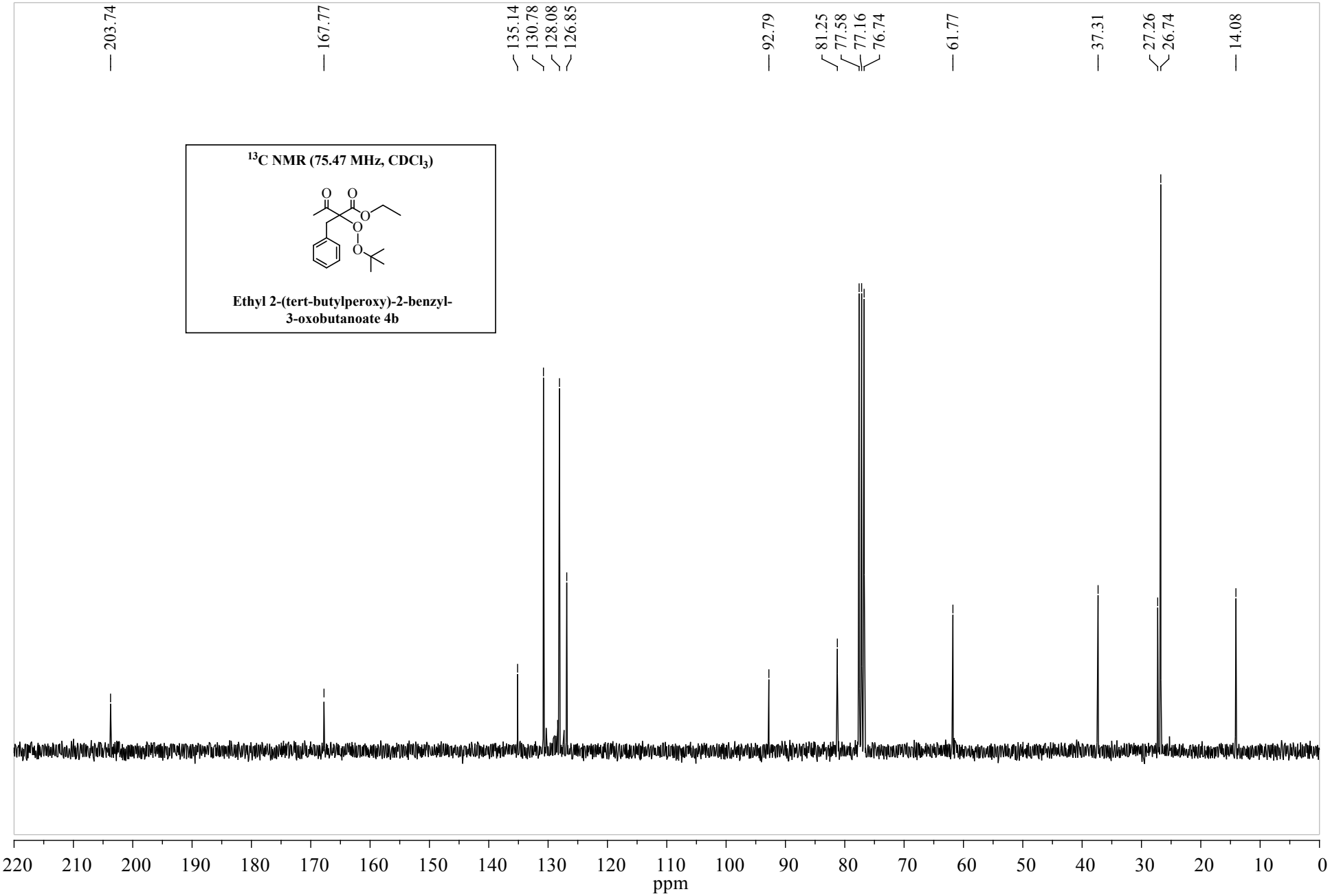

7.28
7.26
7.05
7.03
3.52
2.41
2.27
2.03
$^1$H NMR (300.13 MHz, $CDCl_3$)
3-benzyl-3-{[(2,4-dioxopentan-3-ylidene)amino]oxy}pentane-2,4-dione 3o
3.16
2.01
2.00
3.00
3.01
5.99
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

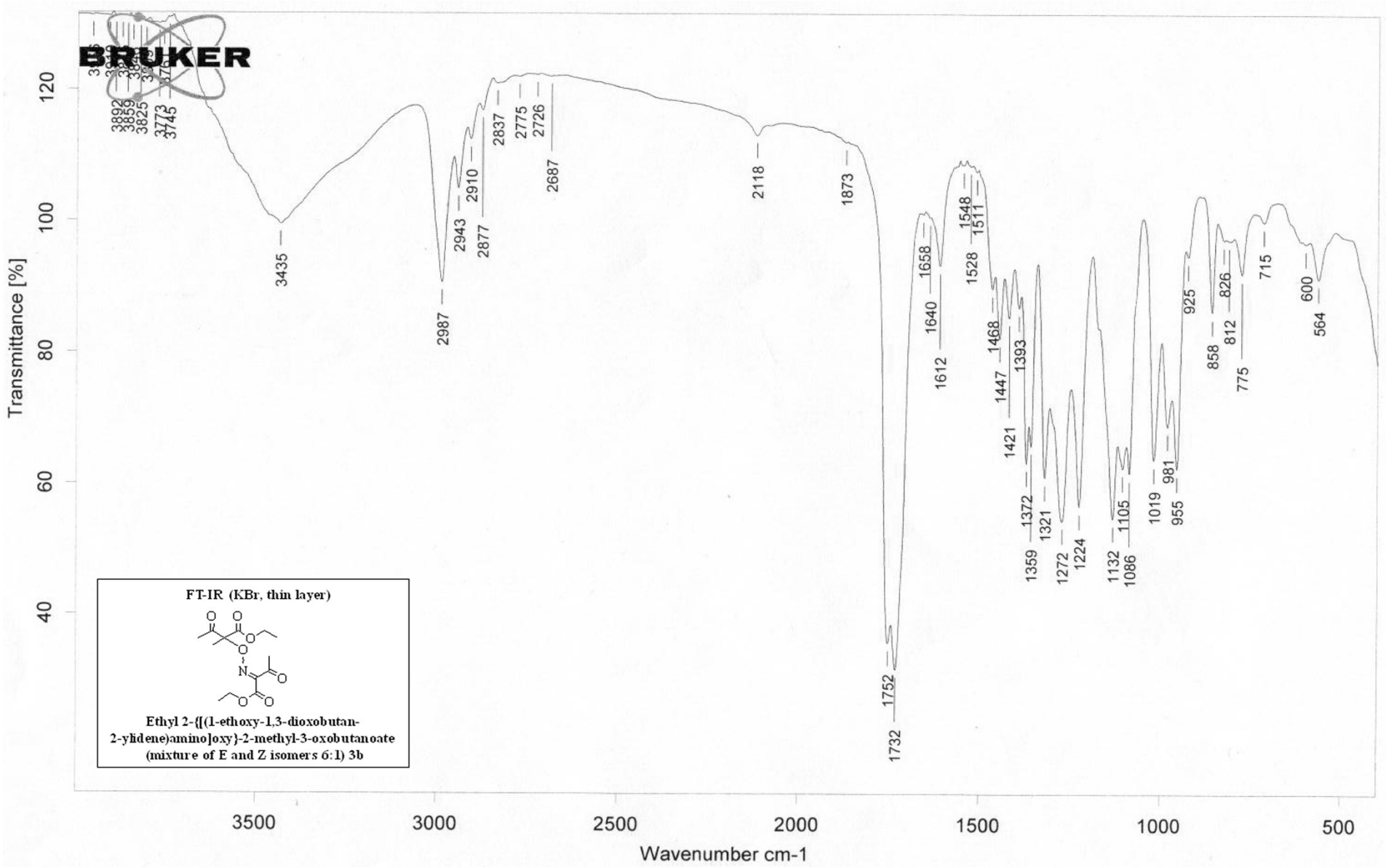

201.41
197.32
193.55
158.03
133.79
130.21
128.71
127.59
99.54
77.58
77.16
76.74
39.07
30.06
27.42
26.01
$^{13}$C NMR (75.47 MHz, CDCl$_3$)
3-benzyl-3-{[(2,4-dioxopentan-3-ylidene)amino]oxy}pentane-2,4-dione 3o
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm

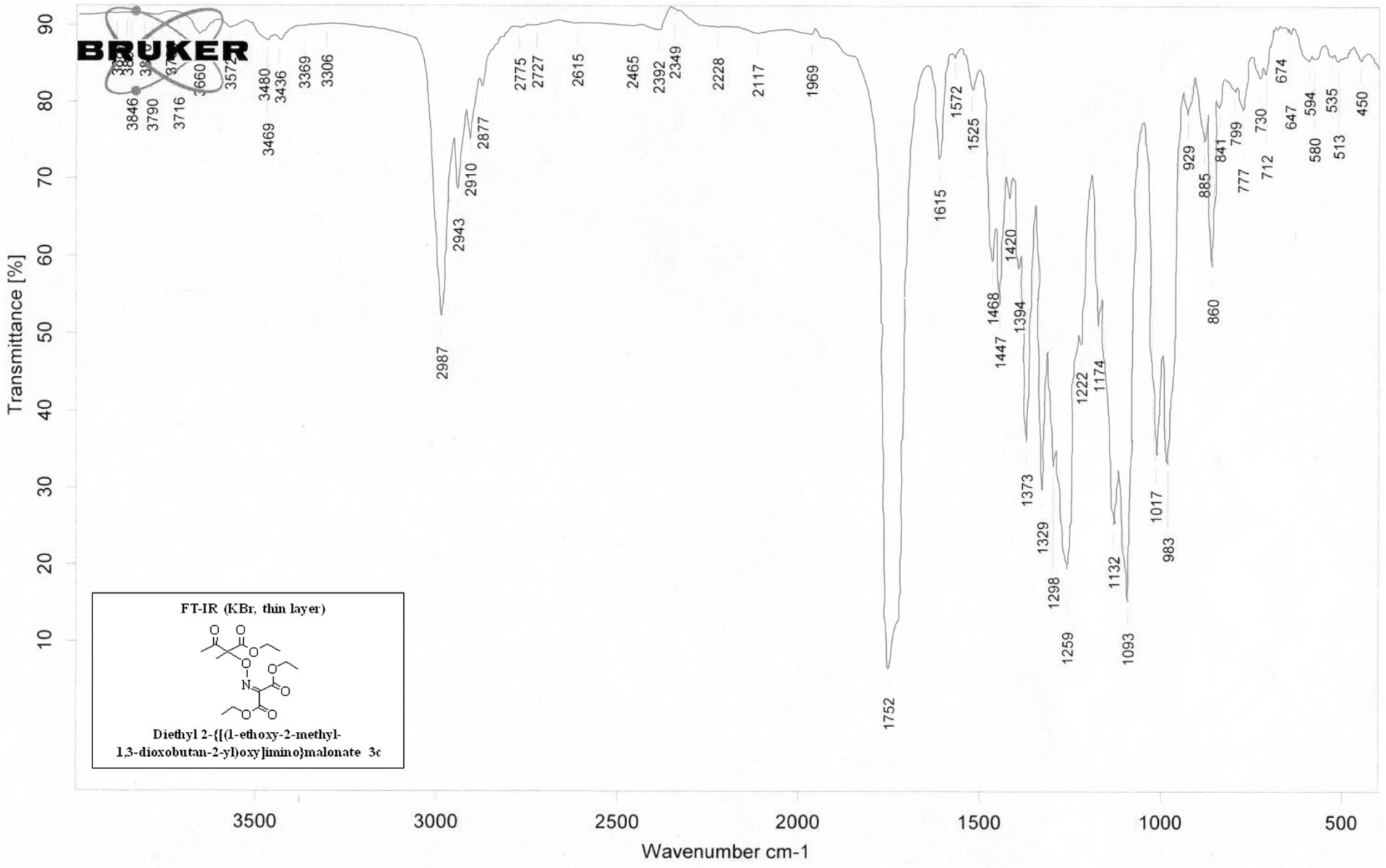

7.26
4.23
4.21
4.18
4.16
2.27
1.56
1.28
1.25
1.24
1.23
$^1$H NMR (300.13 MHz, $CDCl_3$)
Ethyl 2-(tert-butylperoxy)-2-methyl-3-oxobutanoate 4a
2.00
3.00
3.11
12.26
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

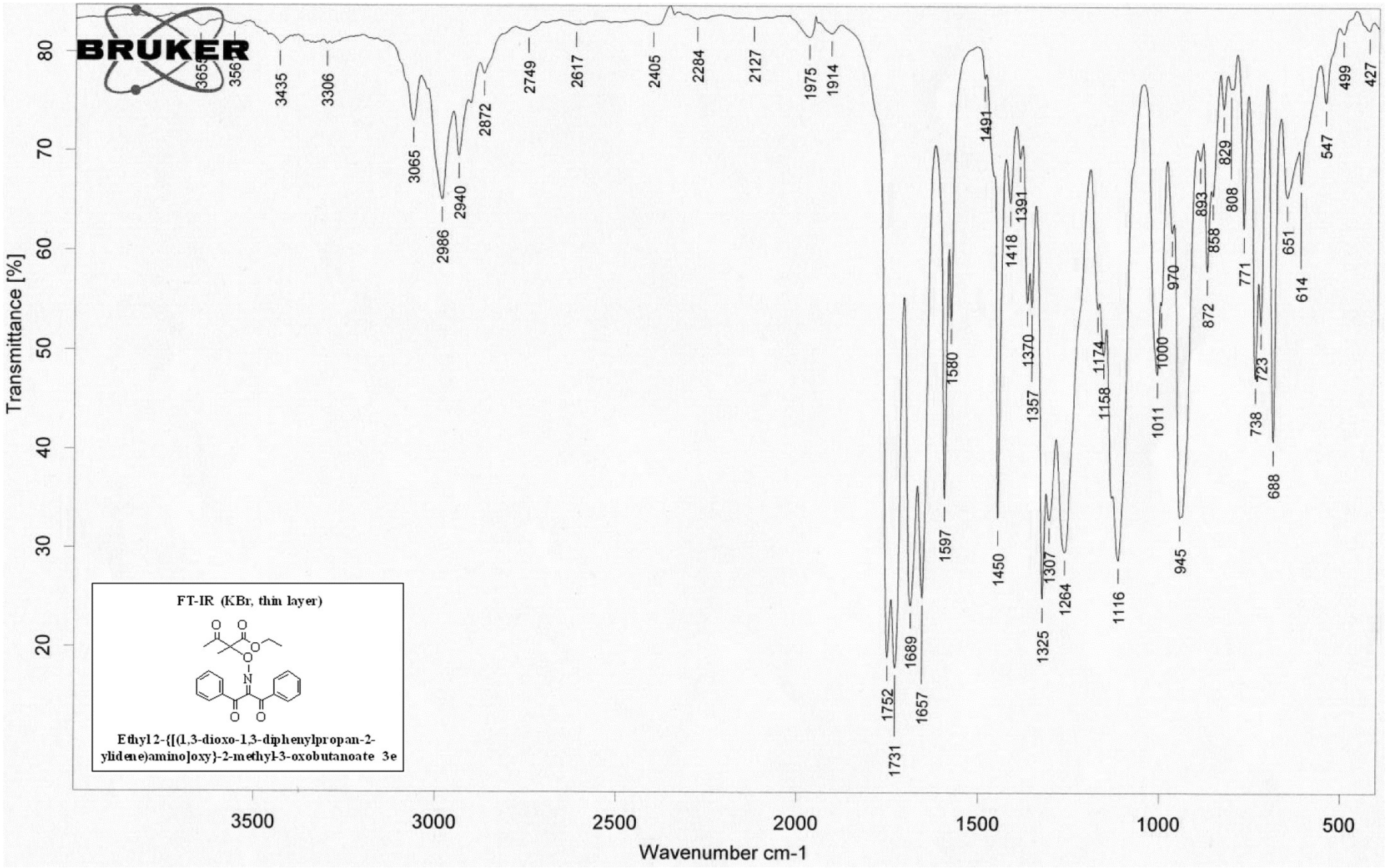

203.82
168.15
89.79
80.84
77.58
77.16
76.74
61.79
26.53
25.74
18.41
14.10
13C NMR (75.47 MHz, CDCl3)
Ethyl 2-(tert-butylperoxy)-2-methyl-
3-oxobutanoate 4a
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

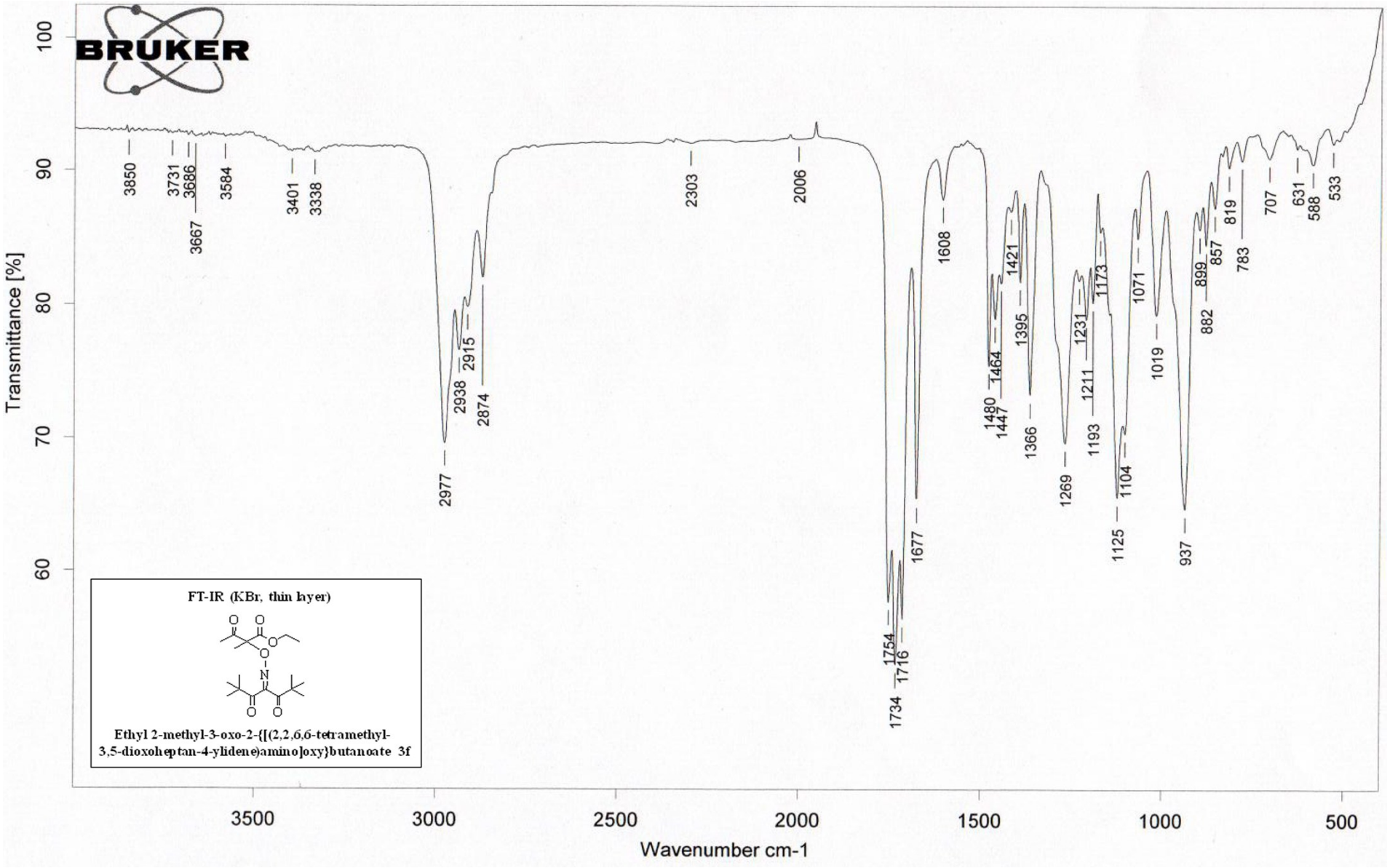

7.26
7.23
4.22
4.21
4.19
4.19
4.17
4.16
4.15
4.14
3.60
3.55
3.36
3.32
1.90
1.32
1.26
1.23
1.21
$^1$H NMR (300.13 MHz, $CDCl_3$)
Ethyl 2-(tert-butylperoxy)-2-benzyl-3-oxobutanoate 4b
5.13
2.07
1.03
1.05
3.00
9.13
3.24
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

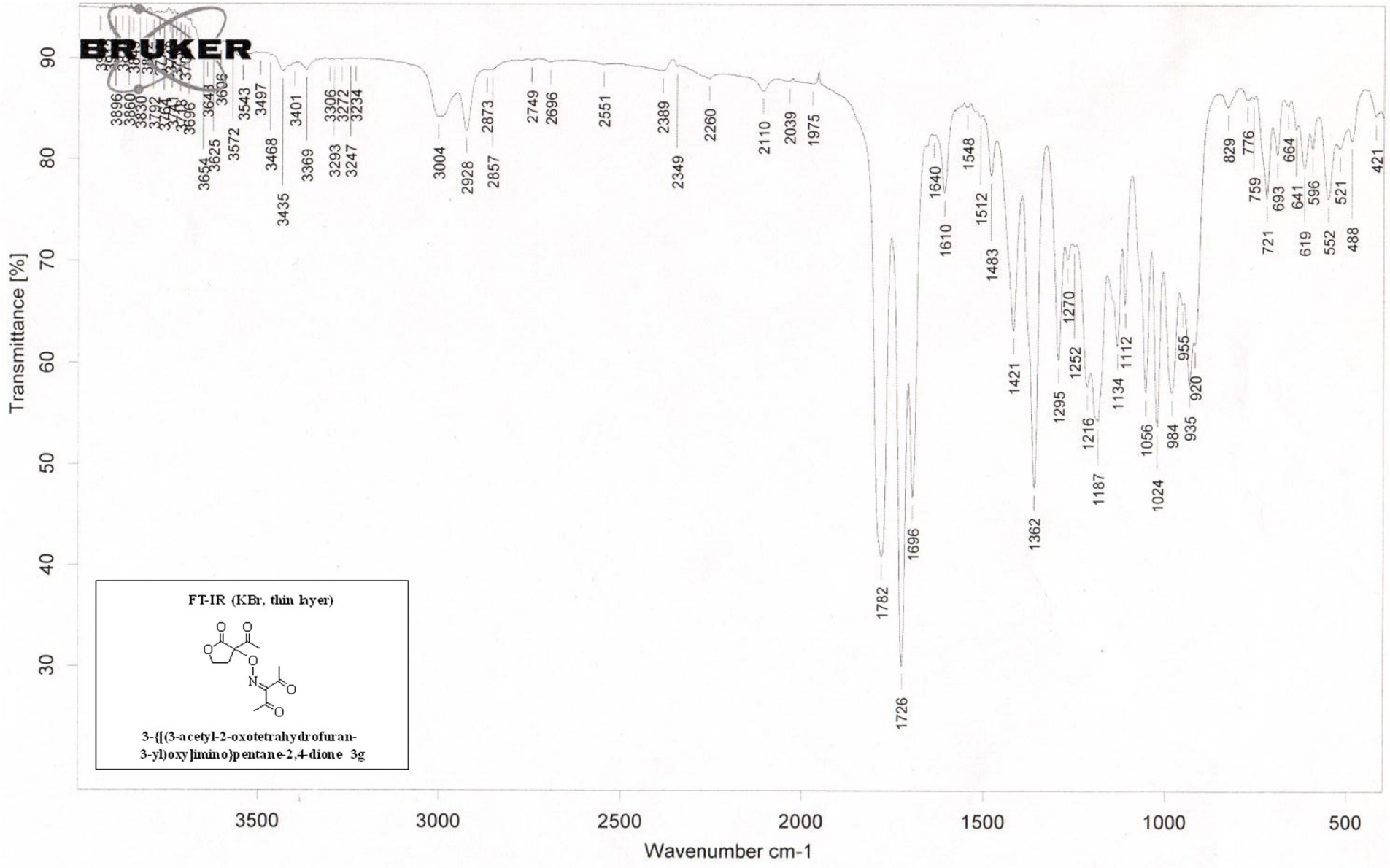

203.74
167.77
135.14
130.78
128.08
126.85
92.79
81.25
77.58
77.16
76.74
61.77
37.31
27.26
26.74
14.08
$^{13}$C NMR (75.47 MHz, CDCl$_3$)
Ethyl 2-(tert-butylperoxy)-2-benzyl-3-oxobutanoate 4b
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

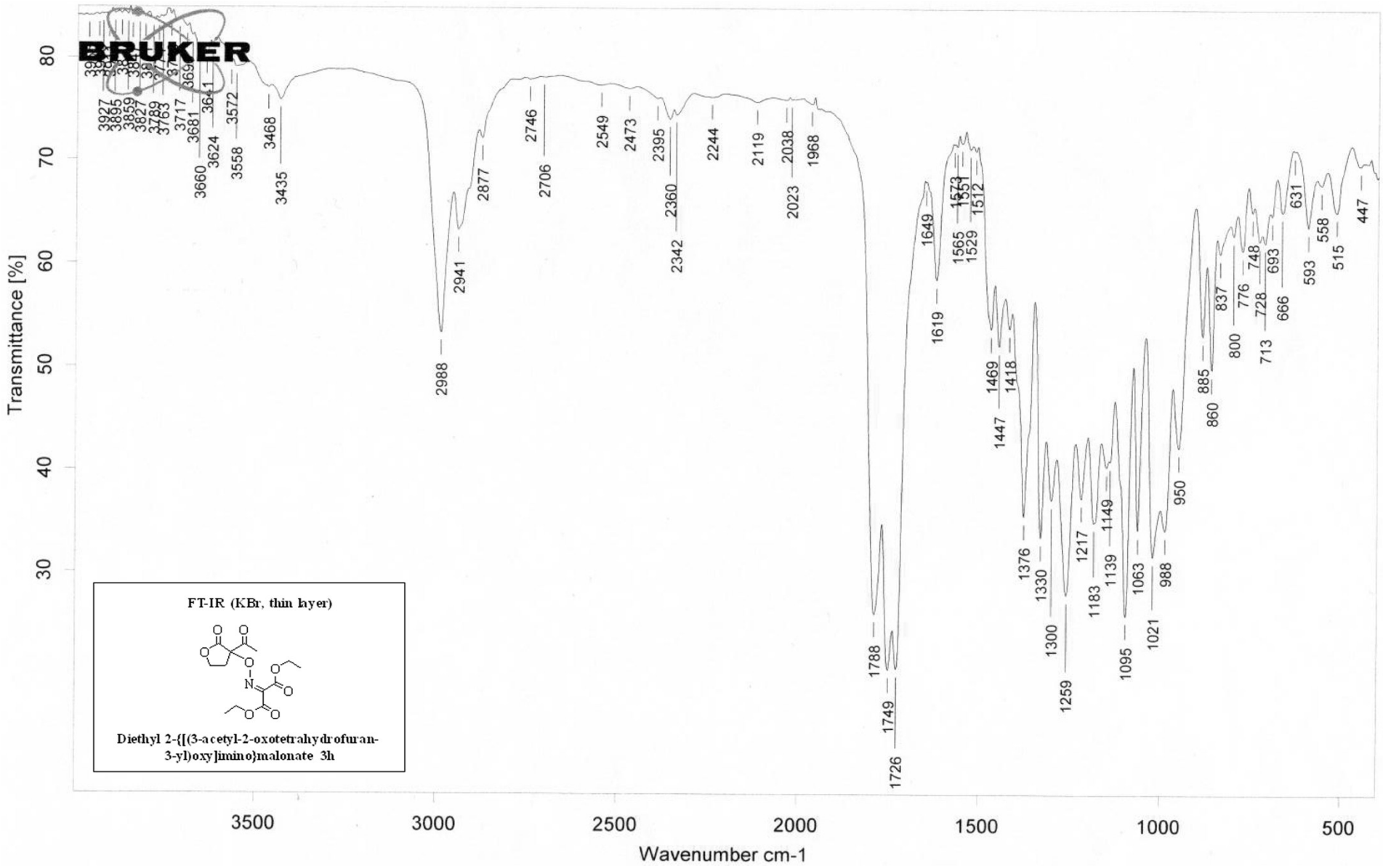

BRUKER
Transmittance [%]
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
120
100
80
60
40
3892
3859
3825
3773
3745
3435
2987
2943
2910
2877
2837
2775
2726
2687
2118
1873
1752
1732
1658
1640
1612
1548
1528
1511
1468
1447
1421
1393
1372
1359
1321
1272
1224
1132
1105
1086
1019
981
955
925
858
826
812
775
715
600
564
FT-IR (KBr, thin layer)
Ethyl 2-{[(1-ethoxy-1,3-dioxobutan-2-ylidene)amino]oxy}-2-methyl-3-oxobutanoate (mixture of E and Z isomers 6:1) 3b

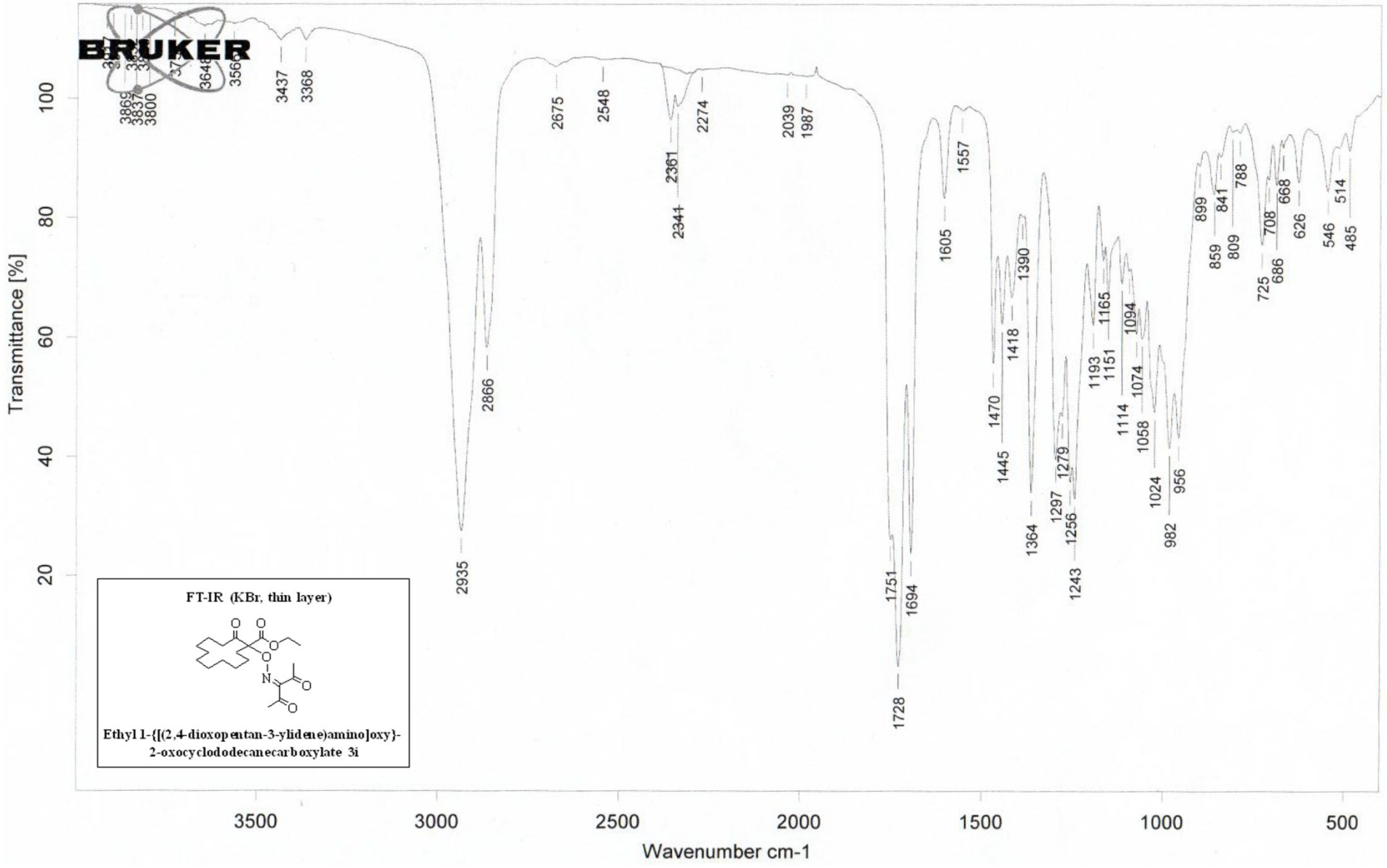

BRUKER
Transmittance [%]
Wavenumber cm-1
FT-IR (KBr, thin layer)
Diethyl 2-{[(1-ethoxy-2-methyl-1,3-dioxobutan-2-yl)oxy]imino}malonate 3c
3846
3790
3716
3660
3572
3480
3469
3436
3369
3306
2987
2943
2910
2877
2775
2727
2615
2465
2392
2349
2228
2117
1969
1752
1615
1572
1525
1468
1447
1420
1394
1373
1329
1298
1259
1222
1174
1132
1093
1017
983
929
885
860
841
799
777
730
712
674
647
594
580
535
513
450

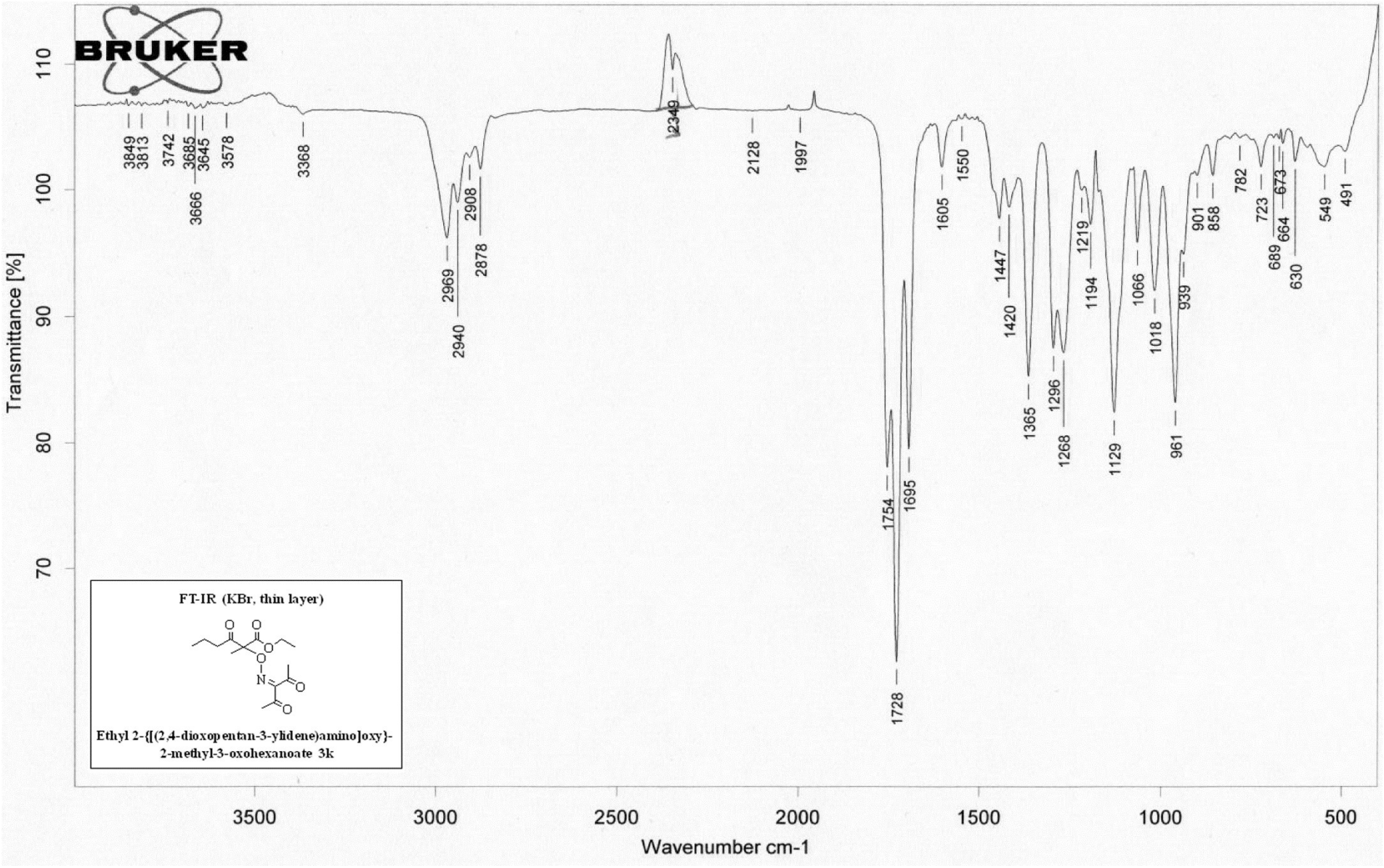

BRUKER
Transmittance [%]
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
20
30
40
50
60
70
80
3655
3565
3435
3306
3065
2986
2940
2872
2749
2617
2405
2284
2127
1975
1914
1752
1731
1689
1657
1597
1580
1491
1450
1418
1391
1370
1357
1325
1307
1264
1174
1158
1116
1011
1000
970
945
893
872
858
829
808
771
738
723
688
651
614
547
499
427
FT-IR (KBr, thin layer)
Ethyl 2-{[(1,3-dioxo-1,3-diphenylpropan-2-ylidene)amino]oxy}-2-methyl-3-oxobutanoate 3e

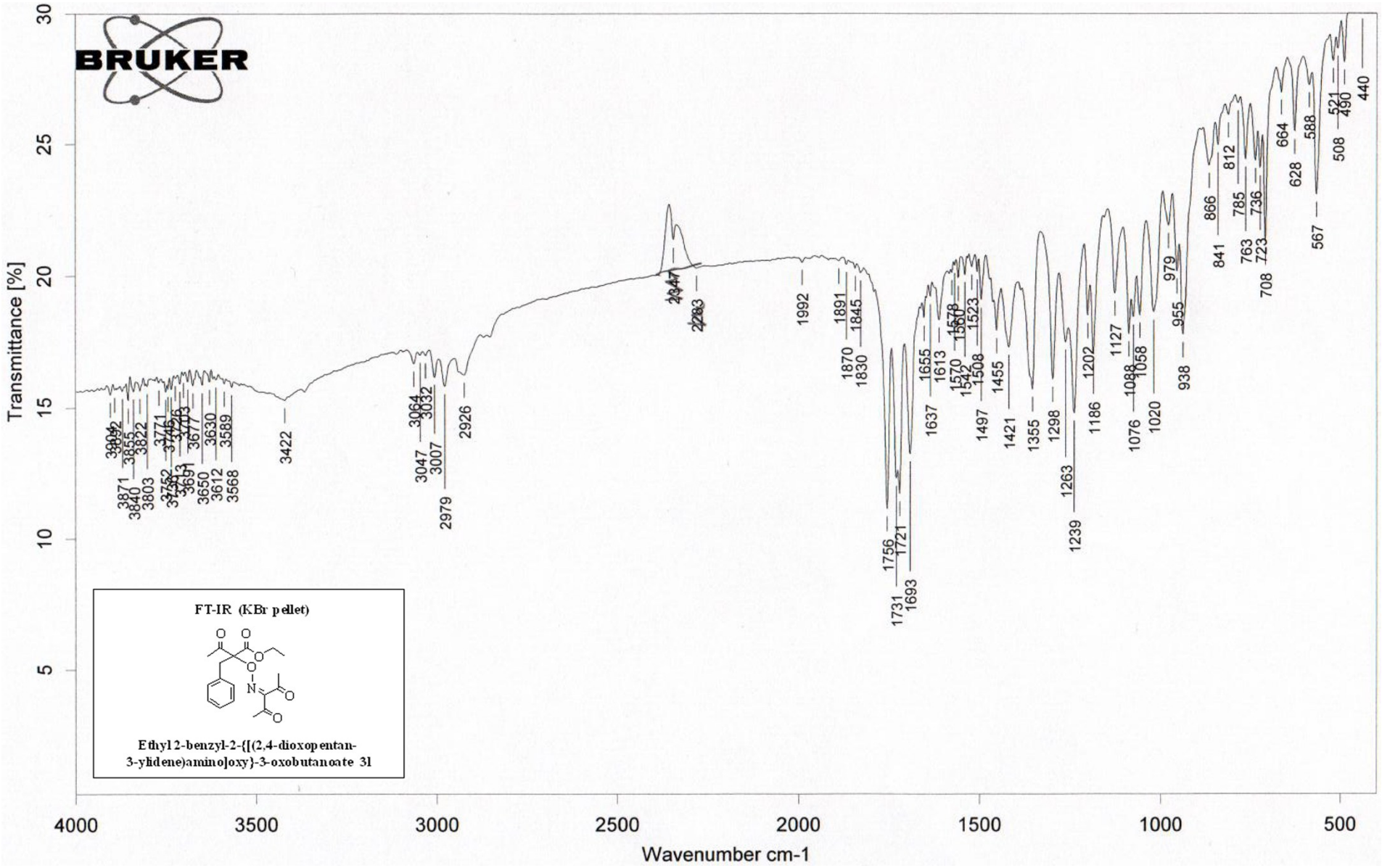

BRUKER
Transmittance [%]
100
90
80
70
60
3850
3731
3686
3667
3584
3401
3338
2977
2938
2915
2874
2303
2006
1754
1734
1716
1677
1608
1480
1464
1447
1421
1395
1366
1269
1231
1211
1193
1173
1125
1104
1071
1019
937
899
882
857
819
783
707
631
588
533
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1
FT-IR (KBr, thin layer)
Ethyl 2-methyl-3-oxo-2-{[(2,2,6,6-tetramethyl-3,5-dioxoheptan-4-ylidene)amino]oxy}butanoate 3f

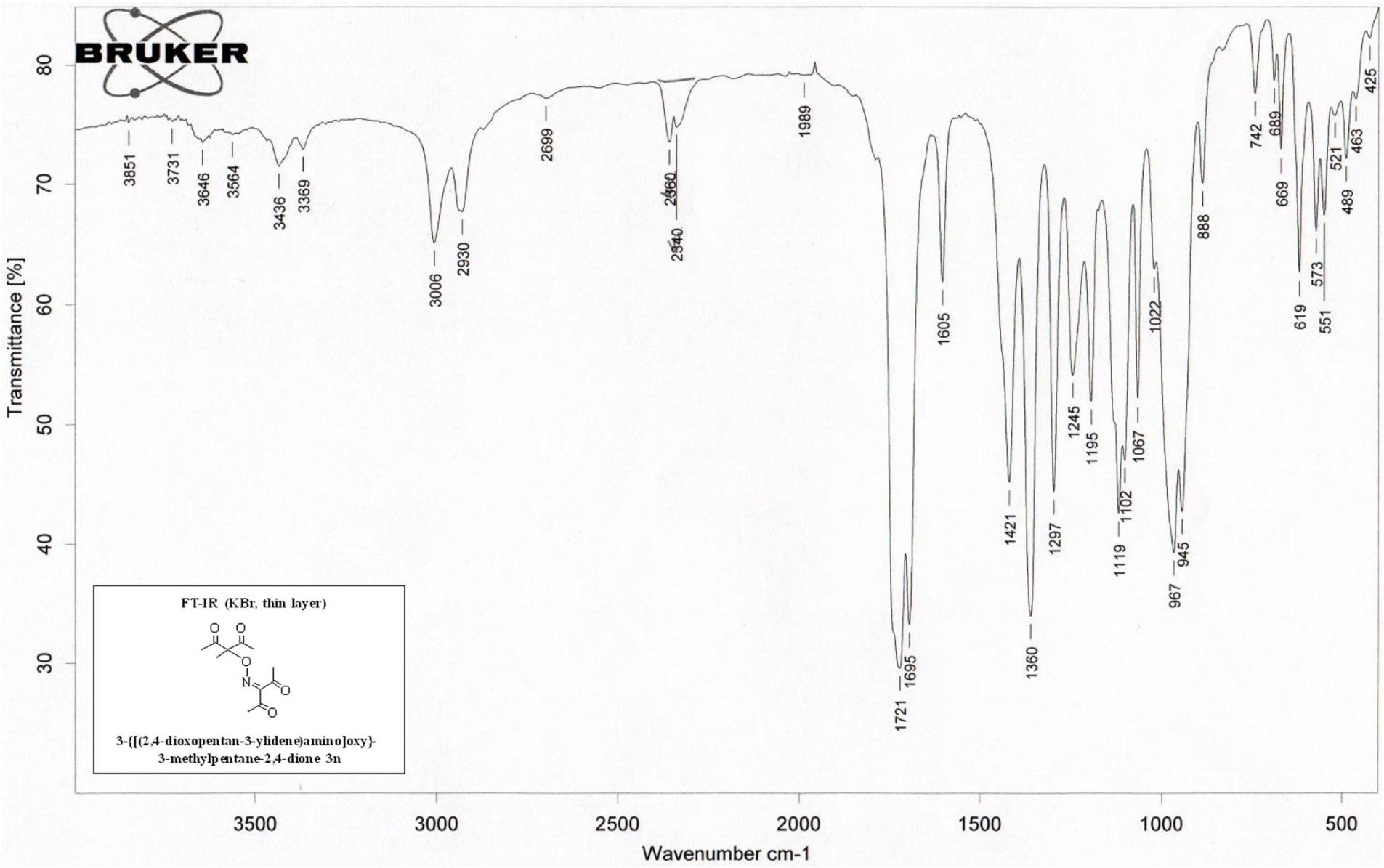

BRUKER
Transmittance [%]
Wavenumber cm-1
FT-IR (KBr, thin layer)
3-{[(3-acetyl-2-oxotetrahydrofuran-3-yl)oxy]imino}pentane-2,4-dione 3g
3896
3860
3830
3654
3625
3643
3606
3572
3543
3497
3468
3435
3401
3369
3306
3293
3272
3247
3234
3004
2928
2873
2857
2749
2696
2551
2389
2349
2260
2110
2039
1975
1782
1726
1696
1640
1610
1548
1512
1483
1421
1362
1295
1270
1252
1216
1187
1134
1112
1056
1024
984
955
935
920
829
776
759
721
693
664
641
619
596
552
521
488
421

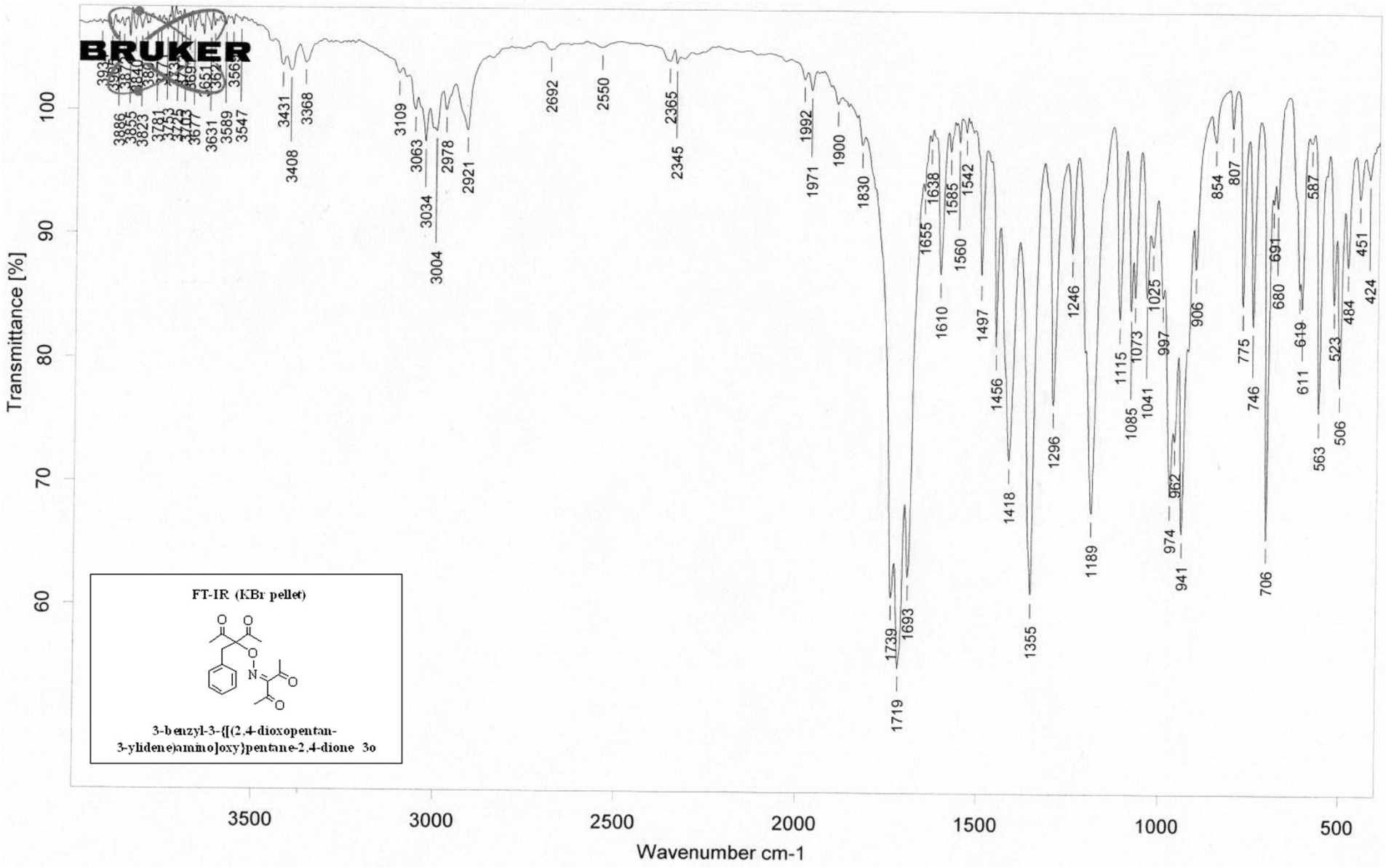

BRUKER
Transmittance [%]
Wavenumber cm-1
FT-IR (KBr, thin layer)
Diethyl 2-{[(3-acetyl-2-oxotetrahydrofuran-3-yl)oxy]imino}malonate 3h

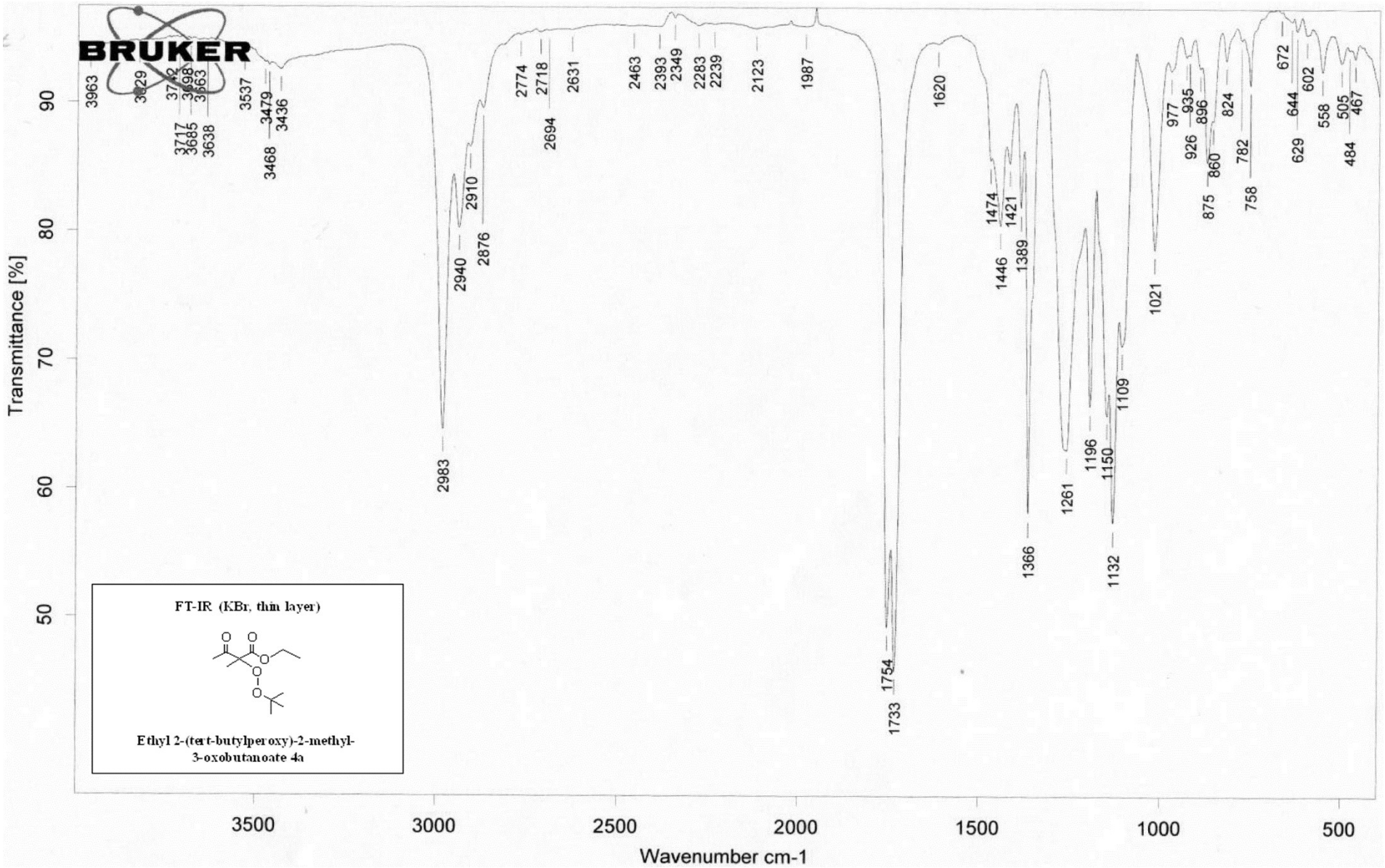

BRUKER
Transmittance [%]
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
20
40
60
80
100
3869
3837
3800
3437
3368
2675
2548
2361
2341
2274
2039
1987
2935
2866
1751
1728
1694
1605
1557
1470
1445
1418
1390
1364
1297
1279
1256
1243
1193
1165
1151
1114
1094
1074
1058
1024
982
956
899
859
841
809
788
725
708
686
668
626
546
514
485
FT-IR (KBr, thin layer)
Ethyl 1-{[(2,4-dioxopentan-3-ylidene)amino]oxy}-2-oxocyclododecanecarboxylate 3i


BRUKER
Transmittance [%]
110
100
90
80
70
3849
3813
3742
3685
3666
3645
3578
3368
2969
2940
2908
2878
2349
2128
1997
1754
1728
1695
1605
1550
1447
1420
1365
1296
1268
1219
1194
1129
1066
1018
961
939
901
858
782
723
689
664
673
630
549
491
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1
FT-IR (KBr, thin layer)
Ethyl 2-{[(2,4-dioxopentan-3-ylidene)amino]oxy}-
2-methyl-3-oxohexanoate 3k


BRUKER
Transmittance [%]
Wavenumber cm-1
4000
3500
3000
2500
2000
1500
1000
500
FT-IR (KBr pellet)
Ethyl 2-benzyl-2-{[(2,4-dioxopentan-3-ylidene)amino]oxy}-3-oxobutanoate 3l


BRUKER
Transmittance [%]
80
70
60
50
40
30
3851
3731
3646
3564
3436
3369
3006
2930
2699
2360
2340
1989
1721
1695
1605
1421
1360
1297
1245
1195
1119
1102
1067
1022
967
945
888
742
689
669
619
573
551
521
489
463
425
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1
FT-IR (KBr, thin layer)
3-{[(2,4-dioxopentan-3-ylidene)amino]oxy}-
3-methylpentane-2,4-dione 3n


BRUKER
FT-IR (KBr pellet)
3-benzyl-3-{[(2,4-dioxopentan-3-ylidene)amino]oxy}pentane-2,4-dione 3o
Transmittance [%]
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
100
90
80
70
60
3886
3855
3823
3781
3677
3631
3589
3547
3431
3408
3368
3109
3063
3034
3004
2978
2921
2692
2550
2365
2345
1992
1971
1900
1830
1739
1719
1693
1655
1638
1610
1585
1560
1542
1497
1456
1418
1355
1296
1246
1189
1115
1085
1073
1041
1025
997
974
962
941
906
854
807
775
746
706
691
680
619
611
587
563
523
506
484
451
424


BRUKER
Transmittance [%]
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
90
80
70
60
50
3963
3629
3717
3685
3638
3537
3479
3468
3436
2983
2940
2910
2876
2774
2718
2694
2631
2463
2393
2349
2283
2239
2123
1987
1754
1733
1620
1474
1446
1421
1389
1366
1261
1196
1150
1132
1109
1021
977
935
926
896
875
860
824
782
758
672
644
629
602
558
505
484
467
FT-IR (KBr, thin layer)
Ethyl 2-(tert-butylperoxy)-2-methyl-3-oxobutanoate 4a