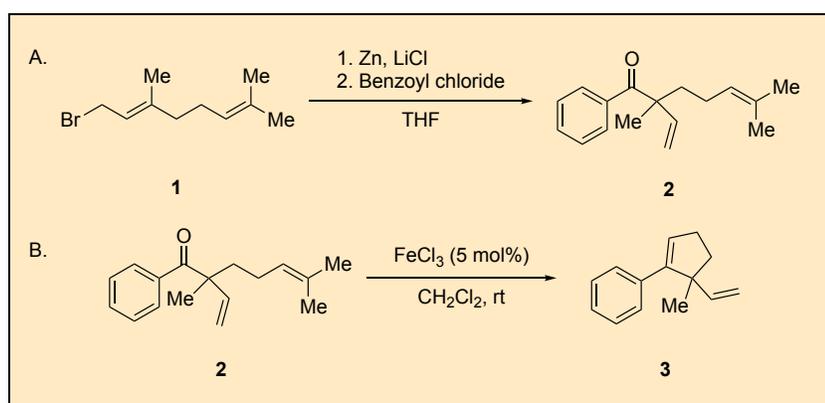


## Carbonyl-Olefin Metathesis for the Synthesis of Cyclic Olefins

Marc R. Becker, Katie A. Rykaczewski, Jacob R. Ludwig, and Corinna S. Schindler\*<sup>1</sup>

Department of Chemistry, University of Michigan, Willard Henry Dow Laboratory, 930 North University Avenue, Ann Arbor, Michigan 48109, USA

Checked by Yang Cao and Kevin Campos



### Procedure (Note 1)

A. *2,6-Dimethyl-1-phenyl-2-vinylhept-5-en-1-one* (2). (Note 2) A flame-dried 500-mL three-necked, round-bottomed flask is fitted with a Teflon-coated magnetic stir bar (egg-shaped, 3.5 cm length x 1.2 cm diameter) and equipped with two rubber septa, one of which is pierced with a needle connected to a nitrogen source. The three-necked flask is charged with activated zinc powder (14.09 g, 215 mmol, 2.5 equiv) (Note 3), lithium chloride (5.11 g, 121 mmol, 1.4 equiv) and anhydrous THF (230 mL). A flame-dried 125-mL pressure-equalizing addition funnel containing a solution of geranyl bromide (21.4 mL, 108 mmol, 1.25 equiv) (Note 4) in

anhydrous THF (20 mL) is attached to the reaction flask and sealed with a rubber septum. The flask is immersed in a 23 °C water bath and the geranyl bromide solution added dropwise over 10 min to the vigorously stirring suspension (Figure 1A). After complete addition, the grey, heterogeneous mixture is stirred for 1.5 h at 23 °C under an atmosphere of nitrogen, at which time stirring is stopped and excess zinc allowed to settle. A flame-dried 500-mL round-bottomed flask is charged with a Teflon-coated magnetic stir bar (egg-shaped, 3.5 cm length x 1.2 cm diameter), benzoyl chloride (**1**) (10.0 mL, 86 mmol, 1.0 equiv.) and anhydrous THF (80 mL) and fitted with a rubber septum with nitrogen inlet and thermometer-probe.



**Figure 1.** A) Preparation of allyl zinc reagent (left); B) Addition of allyl zinc reagent to benzoyl chloride via cannula (right) (photo provided by submitter)

The benzoyl chloride containing flask is immersed in an ice/water bath and the supernatant zinc reagent solution is added via a cannula at a rate that kept the internal temperature below 5-6 °C (Figure 1B). After addition is complete, the flask is maintained in the ice/water bath and the slightly cloudy solution is stirred for 30 min under an atmosphere of nitrogen until full conversion of starting material occurs, as judged by TLC (Note 5). An aqueous saturated  $\text{NH}_4\text{Cl}$  solution (50 mL) and deionized water (50 mL) are added sequentially. After the ice/water bath is removed and the mixture is stirred for 10 min, the biphasic mixture is transferred to a 1-L separatory funnel and the reaction flask rinsed with EtOAc (3 x 10 mL), which is added to the separatory funnel. The aqueous layer is separated, extracted with

EtOAc (3 x 100 mL) and the combined organic layers concentrated under reduced pressure with a rotary evaporator (100 mmHg, bath temperature 40 °C). The oily residue is taken up in EtOAc (200 mL), transferred to a 500-mL separatory funnel and washed with deionized water (2 x 50 mL), an aqueous 1 M NaOH solution (2 x 50 mL) and brine (2 x 50 mL) (Note 6). The organic layer is dried over Na<sub>2</sub>SO<sub>4</sub> (75 g), filtered and concentrated under reduced pressure with a rotary evaporator (100 mmHg, bath temperature 40 °C) and then with a vacuum pump (0.5-1 mmHg) (Note 7). The crude residue is transferred into a 100-mL round-bottomed flask (Note 8) and distilled using a short-path distillation apparatus under vacuum (0.5-1 mmHg) (Figure 2) (Note 9). A forerun (2–3 mL) is collected and discarded, and the fraction distilling at 110 °C is collected to afford aryl ketone **2** (5:1 mixture of olefin isomers) as a pale-yellow oil (18.44 g, 89%) (Notes 10 and 11). Purity of the oil was determined to be 90% by quantitative NMR using dimethyl terephthalate as internal standard (Note 12).



**Figure 2.** Distillation apparatus for the distillation of aryl ketone **2** (photo provided by submitter)

B. (*5-Methyl-5-vinylcyclopent-1-en-1-yl*)benzene (**3**). A flame-dried 500-mL round-bottomed flask is fitted with a Teflon-coated magnetic stir bar (egg-shaped, 3.5 cm length x 1.2 cm diameter) and charged with iron(III) chloride (502 mg, 3.1 mmol, 0.05 equiv) (Note 13), which is immediately suspended in anhydrous CH<sub>2</sub>Cl<sub>2</sub> (230 mL) (Notes 14 and 15).



**Figure 3.** Color change before (left) and after (right) addition of aryl ketone **2** to  $\text{FeCl}_3$  suspension (photo provided by submitter)

The flask is equipped with a flame-dried 125-mL pressure-equalizing addition funnel containing a solution of aryl ketone **2** (15.00 g, 61.9 mmol, 1.0 equiv) (Note 16) in anhydrous  $\text{CH}_2\text{Cl}_2$  (20 mL) and the apparatus sealed with a rubber septum with nitrogen inlet. The aryl ketone solution is added to the suspension of  $\text{FeCl}_3$  over 2 min and the resulting dark-brown solution stirred at 23 °C for 2.5 h (Figure 3), which resulted in full consumption of starting material as determined by TLC analysis (Note 17). The reaction mixture is passed through a short silica plug (180–200 g silica gel equilibrated in  $\text{CH}_2\text{Cl}_2$  in a glass column with 8.5 cm diameter) positioned over a 2-L round-bottomed flask, and the plug is rinsed with  $\text{CH}_2\text{Cl}_2$  (500 mL) applying a brief positive pressure to move the solution through the silica gel (Figure 4).

The eluent is concentrated under reduced pressure with a rotary evaporator (100 mmHg, bath temperature 40 °C) and then with a vacuum pump (0.5–1 mmHg). The residual orange oil is transferred to a 50-mL round-bottomed flask and distilled using a short-path distillation apparatus under vacuum (0.5–1 mmHg) (Note 18). The fraction distilling at 87–91 °C is collected to afford cyclopentene **3** as a colorless oil (10.45 g, 93%) (Notes 19 and 20). Purity of the oil was determined to be 97% by quantitative NMR using dimethyl terephthalate as an internal standard (Note 21).

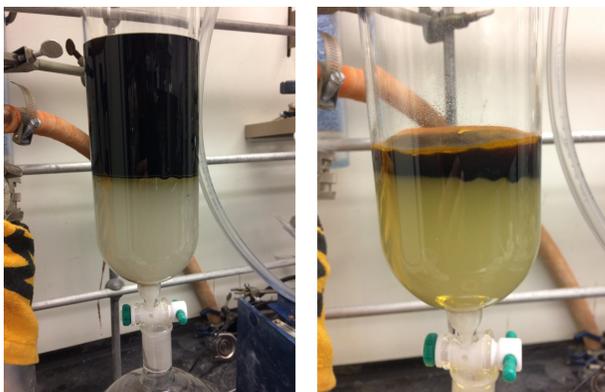


Figure 4. Silica plug before (left) and after (right) filtration and rinsing (photo provided by submitter)

## Notes

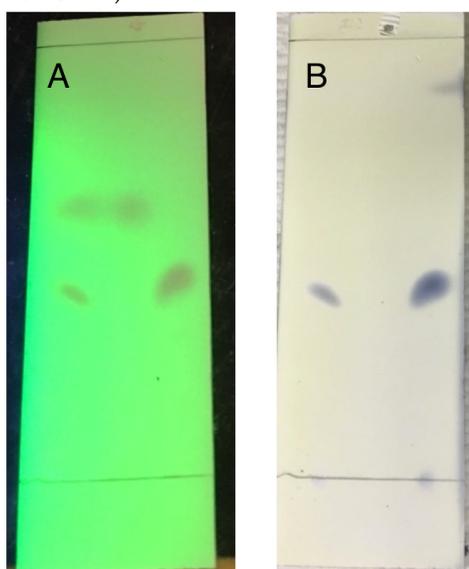
1. Prior to performing each reaction, a thorough hazard analysis and risk assessment should be carried out with regard to each chemical substance and experimental operation on the scale planned and in the context of the laboratory where the procedures will be carried out. Guidelines for carrying out risk assessments and for analyzing the hazards associated with chemicals can be found in references such as Chapter 4 of "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011; the full text can be accessed free of charge at <https://www.nap.edu/catalog/12654/prudent-practices-in-the-laboratory-handling-and-management-of-chemical>. See also "Identifying and Evaluating Hazards in Research Laboratories" (American Chemical Society, 2015) which is available via the associated website "Hazard Assessment in Research Laboratories" at <https://www.acs.org/content/acs/en/about/governance/committees/chemicalsafety/hazard-assessment.html>. In the case of this procedure, the risk assessment should include (but not necessarily be limited to) an evaluation of the potential hazards associated with geranyl bromide, tetrahydrofuran (THF), hexane, ethyl acetate (EtOAc), sodium sulfate ( $\text{Na}_2\text{SO}_4$ ), zinc (Zn), lithium chloride (LiCl), benzoyl chloride, ammonium chloride ( $\text{NH}_4\text{Cl}$ ), sodium hydroxide (NaOH), sodium

- chloride (NaCl), iron(III) chloride ( $\text{FeCl}_3$ ), silica and dichloromethane ( $\text{CH}_2\text{Cl}_2$ ), as well as the proper procedures for vacuum distillation.
- The procedure for the synthesis of aryl ketone **2** was adapted with small modifications from a report by Sämam *et al.*<sup>2</sup>
  - Zinc powder (99.3%) was obtained from Fischer Chemical and activated prior to use. For the reported scale, a 250-mL Erlenmeyer flask, fitted with a Teflon-coated magnetic stir bar (5 cm length  $\times$  1 cm diameter), was charged with zinc powder (~20 g) and 1 M HCl (aq., 75 mL). The suspension was stirred vigorously for 10 min, then filtered with a Büchner funnel and subsequently washed with 1 M HCl (aq., 2  $\times$  50 mL), water (1  $\times$  50 mL), ethanol (2  $\times$  50 mL) and diethyl ether (2  $\times$  50 mL) and the activated zinc powder dried under reduced pressure with a nitrogen atmosphere (Figure 5). Lithium chloride (99%, for molecular biology) was obtained from Acros Organics and dried prior to use at 200 °C under reduced pressure with a vacuum pump. Benzoyl chloride (99%) was obtained from Sigma Aldrich and distilled prior to use. Tetrahydrofuran was obtained from Fisher (Optima) and dried by being passed through a column of activated alumina under argon (using a JC-Meyer Solvent Systems).



Figure 5. Drying activated zinc

- Geranyl bromide (96%) was obtained from Alfa Aesar and distilled prior to use, or purchased from Aldrich (94.6wt% by QNMR) and used as received. The submitters observed that distilled geranyl bromide (colorless oil) turned orange within a few days indicating decomposition. Alternatively, the submitters found that geranyl bromide can be prepared from geraniol following a procedure by Baer *et al.*<sup>3</sup>, which, when performed on a 30 g scale, afforded geranyl bromide in 83-86% yield and 97wt% purity after vacuum distillation.
- The reaction progress was followed by TLC analysis on silica gel with 98:2 hexane/EtOAc as eluent and visualization with UV (254 nm) and ceric ammonium molybdate (CAM) stain. The starting material benzoyl chloride **1** has  $R_f = 0.59$  (no CAM activity) and the ketone product **2** has  $R_f = 0.41$  (blue with CAM).



**Figure 6.** TLC analysis at the end of the reaction and visualized with A) UV light, and B) ceric ammonium molybdate solution

- A basic wash was necessary to remove trace amounts of benzoic acid to avoid precipitation of evaporated benzoic acid in the distillation apparatus during distillation. A basic wash without prior removal of THF proved to be ineffective.

7. An aliquot (29.8 mg) of the crude material was taken and the purity assessed as 93wt% by quantitative  $^1\text{H}$  NMR using dimethyl terephthalate as internal standard.
8. The flask used for the distillation needs to be free from any Lewis acid impurities to avoid undesired side reactions at the high temperatures employed during the distillation. Therefore, a wash with strong acid (e.g. concentrated hydrochloric acid) is suggested.
9. It is suggested to apply vacuum carefully while stirring vigorously to prevent bumping. When the desired product started to distill, the distillation head was mantled in aluminum foil to keep the head temperature constant.
10. The checkers reported that product **2** was obtained as a 5:1 mixture of inseparable olefin isomers. The checkers reported that use of a lower distillation temperature (110 °C versus 125 °C for the submitters) led to a reduced appearance of the minor isomer. Compound **2** is characterized as follows:  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  : 1.40 (s, 3H), 1.47 (s, 3H), 1.65 (s, 3H), 1.78 (d,  $J = 8.4$  Hz, 2H), 1.96 (d,  $J = 8.2$  Hz, 2H), 5.05 (s, 1H), 5.18–5.32 (m, 2H), 6.19 (dd,  $J = 17.6, 10.7$  Hz, 1H), 7.39 (t,  $J = 7.7$  Hz, 2H), 7.47 (t,  $J = 7.4$  Hz, 1H), 7.86 (d,  $J = 7.8$  Hz, 2H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  : 17.4, 22.9, 23.0, 25.6, 39.0, 53.6, 114.7, 124.0, 127.9, 129.0, 131.5, 131.9, 137.8, 143.2, 204.7. IR (film): 2968, 2914, 1677, 1630, 1445, 1411, 1375, 1222, 1175, 1001, 964, 916, 828, 792, 717, 693, 664  $\text{cm}^{-1}$ . HRMS: calcd for  $\text{C}_{17}\text{H}_{23}\text{O}^+$  [ $\text{M} + \text{H}$ ] $^+$ : 243.1743; Found: 243.1756.
11. A second run at identical scale provided 18.27 g (90wt%, 79% yield) of the same colorless oil.
12. The purity was assessed as 90wt% by quantitative  $^1\text{H}$  NMR using dimethyl terephthalate as internal standard (the combined integral of the signals at 5.02 and 4.65 ppm was used in reference to the signal at 3.94 ppm of the internal standard). It was observed that the isomer level increases significantly during high temperature vacuum distillation.
13.  $\text{FeCl}_3$  (98%) was obtained from Strem Chemicals and used as received.
14. Anhydrous  $\text{CH}_2\text{Cl}_2$  was obtained from Fisher (Stabilized/Certified ACS) and dried by being passed through a column of activated alumina under argon (using a JC-Meyer Solvent Systems).
15.  $\text{FeCl}_3$  was weighed out onto a metal spatula (1.5 cm width) and washed into the round-bottomed flask using  $\text{CH}_2\text{Cl}_2$ .
16. The isomeric ratio of aryl ketone **2** does not have an impact on the outcome of the carbonyl-olefin metathesis reaction due to isomerization, which occurs under the metathesis conditions; therefore, the isomeric

- purity is not considered in the calculation of the starting material or in the reaction's yield.
- The reaction progress was followed by TLC analysis on silica gel with 19:1 hexane/EtOAc as eluent and visualization with UV (254 nm). The starting material aryl ketone **2** has  $R_f = 0.38$  and the product cyclopentene **3** has  $R_f = 0.62$ . Note: A side product with a similar  $R_f$  value as the starting material was observed by TLC during the course of the reaction. To determine full consumption of starting material, a 0.1 mL aliquot was taken from the reaction mixture, passed through a pipette silica plug (~3 cm length) and eluted with 10 mL  $\text{CH}_2\text{Cl}_2$ . The eluent was concentrated in vacuo and analyzed by  $^1\text{H}$  NMR.
  - The same distillation setup as illustrated in Figure 2 was used. The submitters obtained slightly higher yields, when the flask used to collect the product was immersed in a water/ice bath.
  - The product **3** was characterized as follows:  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  : 1.33 (s, 3H), 1.87–1.93 (m, 1H), 2.00–2.05 (m, 1H), 2.36–2.49 (m, 2H), 5.05–5.12 (m, 2H), 6.05 (d,  $J = 2.6$  Hz, 1H), 6.09 (dd,  $J = 17.5, 10.6$  Hz, 1H), 7.22 (dd,  $J = 8.3, 6.3$  Hz, 1H), 7.25–7.32 (m, 2H), 7.43 (d,  $J = 7.2$  Hz, 2H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  : 23.4, 29.6, 41.9, 52.2, 111.3, 126.7, 127.1, 127.9, 128.8, 137.0, 146.0, 148.9; IR (film): 3081, 3053, 2929, 2844, 1634, 1598, 1491, 1444, 1370, 1297, 1131, 1097, 1075, 1033, 1000, 959, 908, 828, 758, 695, 670  $\text{cm}^{-1}$ . HRMS:  $m/z$  calcd. for  $\text{C}_{14}\text{H}_{17}^+$  [M+H]: 185.1325; Found: 185.1331.
  - A second reaction on identical scale provided 10.46 g (92%) with 96wt% purity.
  - The purity was assessed as 97wt% by quantitative  $^1\text{H}$  NMR using dimethyl terephthalate as internal standard (the average integral of the signals at 7.42, 7.27 and 7.22 ppm was used in reference to the signal at 8.12 ppm of the internal standard).

## Working with Hazardous Chemicals

The procedures in *Organic Syntheses* are intended for use only by persons with proper training in experimental organic chemistry. All hazardous materials should be handled using the standard procedures for work with chemicals described in references such as "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011;

the full text can be accessed free of charge at [http://www.nap.edu/catalog.php?record\\_id=12654](http://www.nap.edu/catalog.php?record_id=12654)). All chemical waste should be disposed of in accordance with local regulations. For general guidelines for the management of chemical waste, see Chapter 8 of Prudent Practices.

In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior to performing a reaction, a thorough risk assessment should be carried out that includes a review of the potential hazards associated with each chemical and experimental operation on the scale that is planned for the procedure. Guidelines for carrying out a risk assessment and for analyzing the hazards associated with chemicals can be found in Chapter 4 of Prudent Practices.

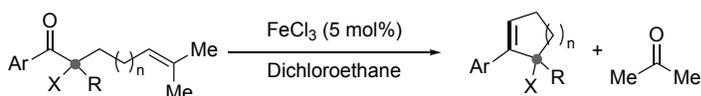
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries or damages claimed to have resulted from or related in any way to the procedures herein.

## Discussion

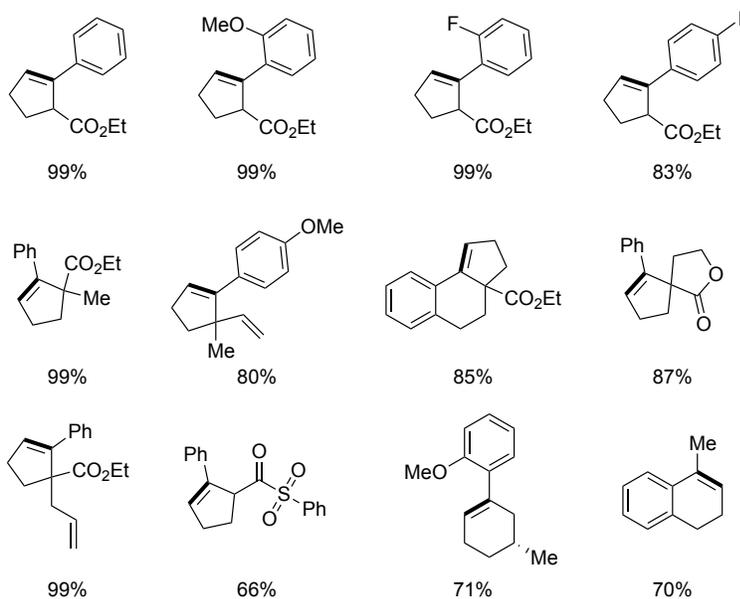
The carbonyl-olefin metathesis reaction is characterized by the exchange of double-bonded atoms in a carbonyl and an olefin to form a new carbonyl and a new olefin. The most common protocol for this reaction involves using precious metal alkylidenes as reagents that provide ring-closing carbonyl olefin metathesis products with a corresponding amount of a catalytically inactive metal-oxo by-product.<sup>4,5</sup> Our lab recently developed a carbonyl-olefin metathesis reaction relying on FeCl<sub>3</sub> as an environmentally benign Lewis acid catalyst that allows for the synthesis of cyclic olefins under mild conditions.<sup>6-8</sup> The reported method is operationally facile and employs catalyst loadings as low as 5 mol%. Notably, the reaction occurs at ambient temperature, utilizes a cheap, abundant metal salt as a catalyst, and produces an easily removable, organic compound as the sole by-product. Table 1 demonstrates a small selection of the broad substrate scope for the

carbonyl-olefin metathesis reaction. Generally, aromatic ketones with electron-donating or withdrawing groups are converted to the corresponding cyclopentene products in good to excellent yields. Furthermore, the reaction protocol allows for efficient access to cyclohexenes and structurally complex motifs such as tricycles and spirocycles.

**Table 1. Scope of the iron(III)-catalyzed carbonyl-olefin metathesis reaction**



*Selected Examples*



\*Conditions: ketone (1 equiv.), FeCl<sub>3</sub> (5 mol%), dichloroethane (0.1-0.01M), rt, 1-24 h.

In this work, we report a carbonyl-olefin metathesis protocol that provides an operationally simple and easily scalable synthesis of cyclic olefin **3**. We demonstrate that the required starting material for the

carbonyl-olefin metathesis can be prepared from readily accessible and cheap reagents allowing for rapid access to large quantities of compound. The carbonyl-olefin metathesis as a key transformation was carried out on 15 g-scale using  $\text{FeCl}_3$  as a cheap and environmentally benign Lewis acid catalyst. Notably, the reported protocol gives the desired product in excellent yield and purity. This manuscript demonstrates the potential of carbonyl-olefin metathesis as an economical and sustainable approach for the synthesis of cyclic olefins.

## References

1. Department of Chemistry, University of Michigan, Willard Henry Dow Laboratory, 930 North University Avenue, Ann Arbor, Michigan 48109, USA. E-mail: [corinnas@umich.edu](mailto:corinnas@umich.edu). This publication is based on work supported by the NIH/National Institute of General Medical Sciences (R01-GM118644), the David and Lucile Packard Foundation and Alfred P. Sloan Foundation (fellowships to C.S.S.). J.R.L. thanks the National Science Foundation for a predoctoral fellowship.
2. Sämman, C.; Knochel, P. *Synthesis* **2013**, *45*, 1870–1876.
3. Baer, P.; Rabe, P.; Citron, C. A.; de Oliveira Mann, C. C.; Kaufmann, N.; Groll, M.; Dickschat, J. S. *ChemBioChem* **2014**, *15*, 213–216.
4. Fu, G.C.; Grubbs, R.H. *J. Am. Chem. Soc.* **1993**, *115*, 3800–3801.
5. Hong, B.; Li, H.; Wu, J.; Zhang, J.; Lei, X. *Angew. Chem. Int. Ed.* **2015**, *54*, 1011–1015.
6. Ludwig, J.R.; Zimmerman, P.M.; Gianino, J.B.; Schindler, C.S. *Nature* **2016**, *533*, 374–379.
7. McAtee, C.C.; Riehl, P.S.; Schindler, C.S. *J. Am. Chem. Soc.* **2017**, *139*, 2960–2963.
8. Ludwig, J.R.; Phan, S.; McAtee, C.C.; Zimmerman, P.M.; Devery III, J.J.; Schindler, C.S. *J. Am. Chem. Soc.* **2017**, *139*, 10832–10842.

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**Appendix**  
**Chemical Abstracts Nomenclature (Registry Number)**

Geranyl bromide: 2,6-Octadiene, 1-bromo-3,7-dimethyl-, (2*E*); (6138-90-5)

Zinc; (7440-66-6)

Lithium chloride; (7447-41-8)

Benzoyl chloride; (98-88-4)

Iron(III) chloride: Iron chloride; (7705-08-0)



Marc R. Becker was born in Germany and received his B.S. and M.S. degree in chemistry at the University of Muenster, Germany. In 2016, he started his graduate studies at the University of Michigan, where he is currently pursuing his Ph.D. under the supervision of Prof. Corinna S. Schindler. His research interests are method development and their application in natural product synthesis.



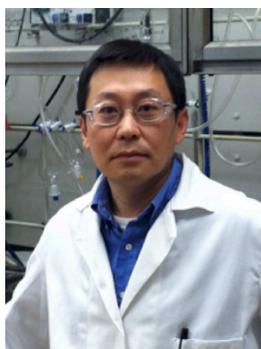
Katie A. Rykaczewski was born in 1996 in Alaska. She is currently enrolled in Seattle University's undergraduate program working towards a B.S. in Biochemistry. She joined the Langenhan group in April 2016 and studies the synthesis of *N*-linked glycoproteins. She was awarded an NSF REU fellowship to work at the University of Michigan under the direction of Prof. Corinna S. Schindler during the summer of 2017.



Jacob R. Ludwig received his B.Sc. in chemistry in 2014 from Michigan State University, where he performed research in the laboratory of Jetze Tepe. After graduation, he joined the Schindler lab at the University of Michigan to pursue his Ph.D. degree.

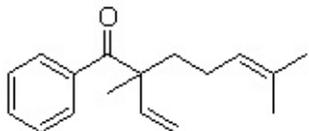


Corinna S. Schindler received her diploma in chemistry from the Technical University of Munich. After a research stay with K.C. Nicolaou at the Scripps Research Institute, she joined the group of Erick Carreira at ETH Zürich for her graduate studies. She then returned to the US to conduct postdoctoral studies with Eric Jacobsen at Harvard before starting her independent career at the University of Michigan in 2013.



Yang Cao received his Ph.D. in medicinal chemistry from University of Toledo. He joined the SPRI in 2007, and is currently working in the Project Chemistry Department of the Process Research, MRL, Rahway, Merck & Co., Inc.

crude aryl ketone  
1H NMR in CDCl<sub>3</sub>  
500 MHz

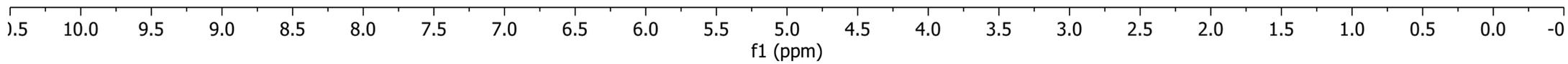


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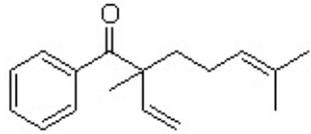
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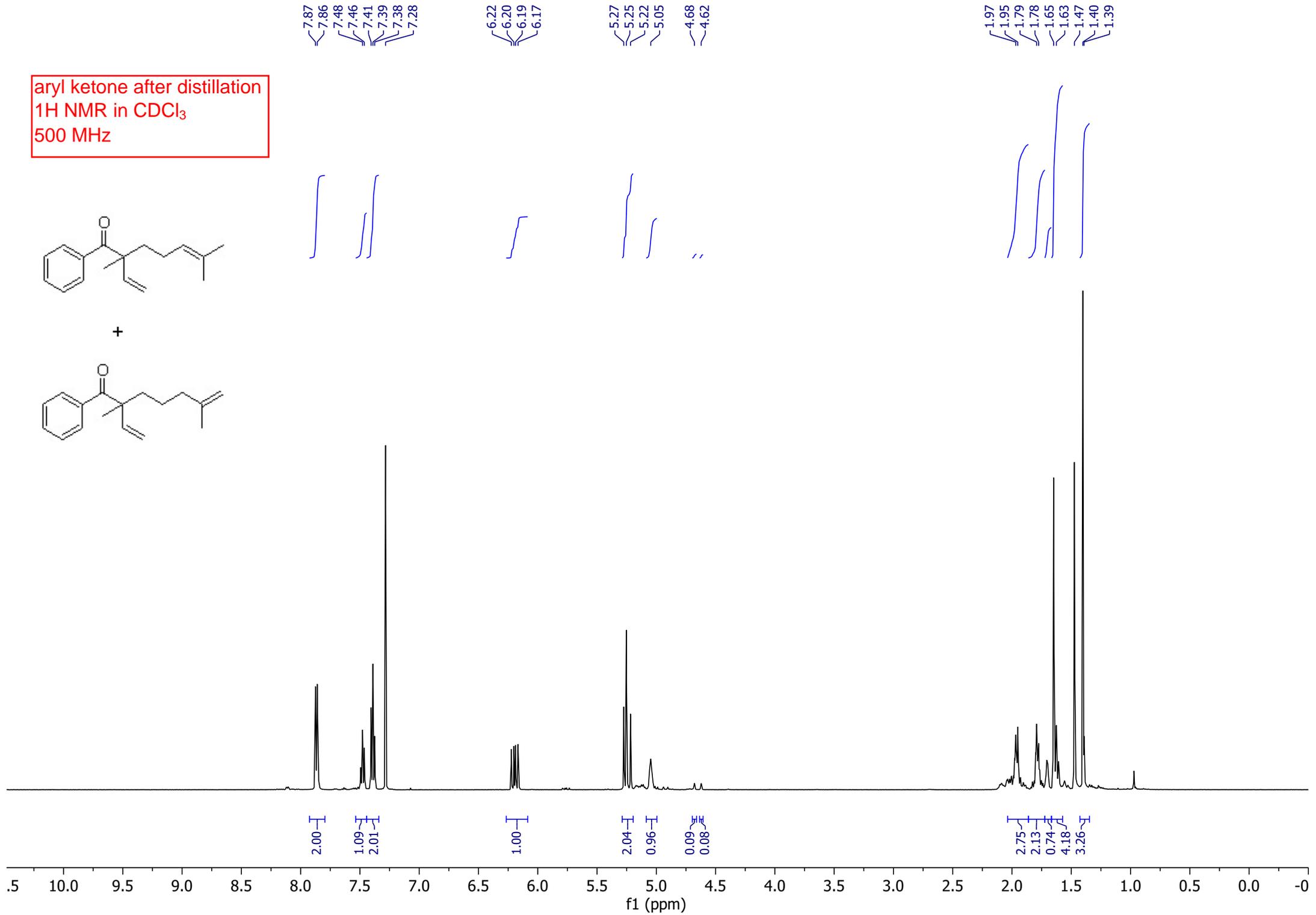
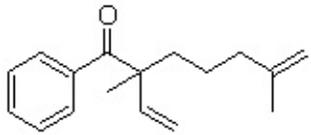
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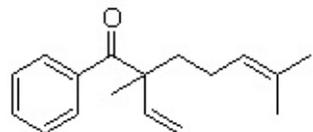
aryl ketone after distillation  
1H NMR in CDCl<sub>3</sub>  
500 MHz



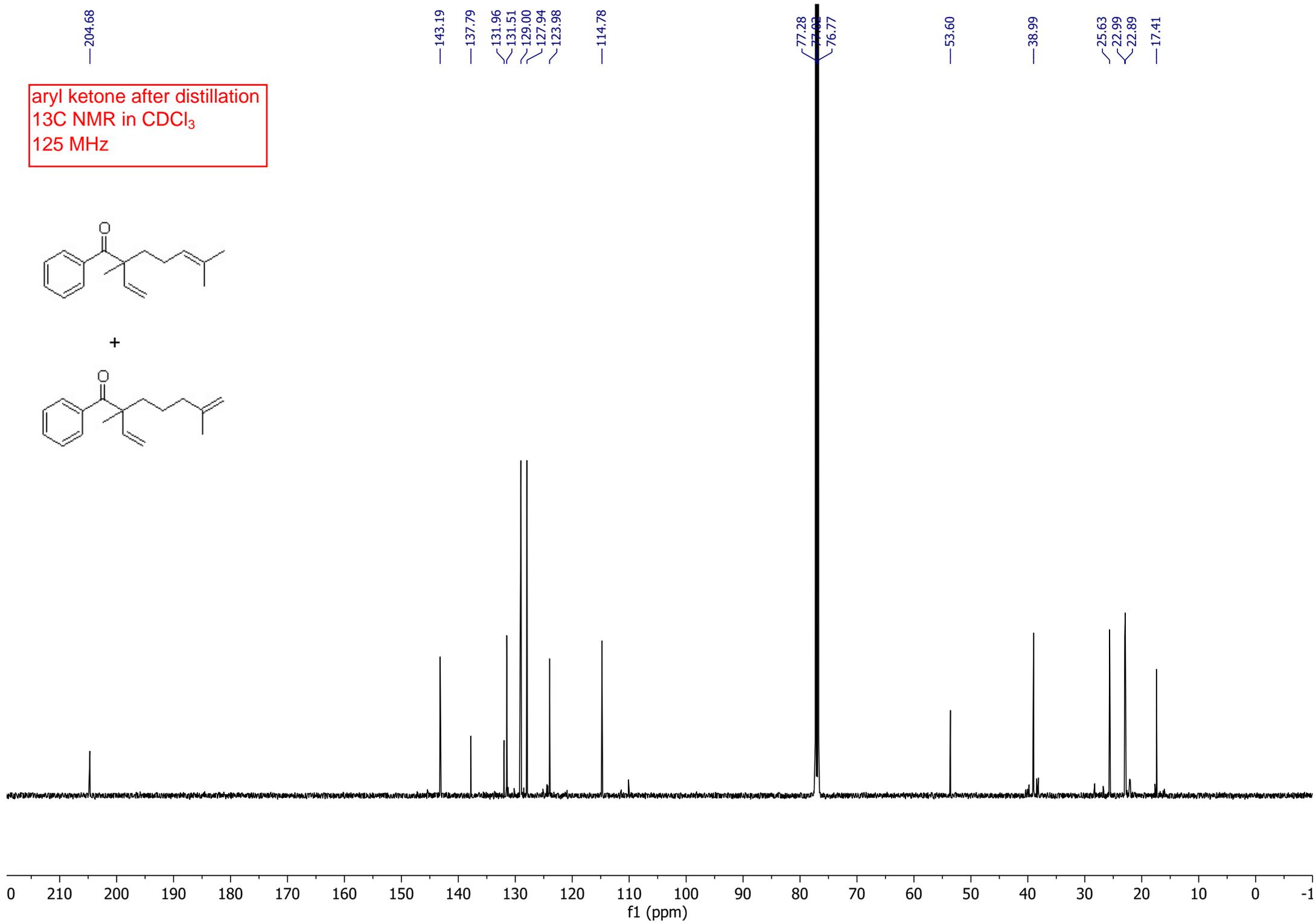
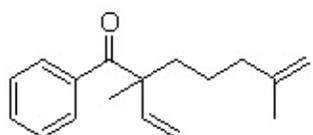
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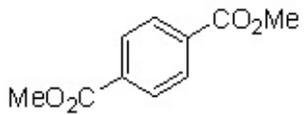
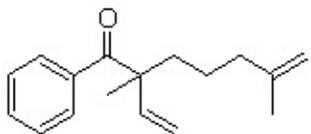
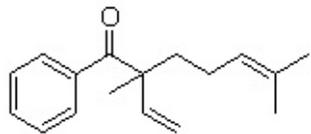
aryl ketone after distillation  
13C NMR in CDCl<sub>3</sub>  
125 MHz



+



aryl ketone after distillation 29.24 mg  
Dimethyl terephthalate (IS) 17.48 mg  
1H NMR in CDCl<sub>3</sub>, 500 MHz



8.12  
7.87  
7.87  
7.86  
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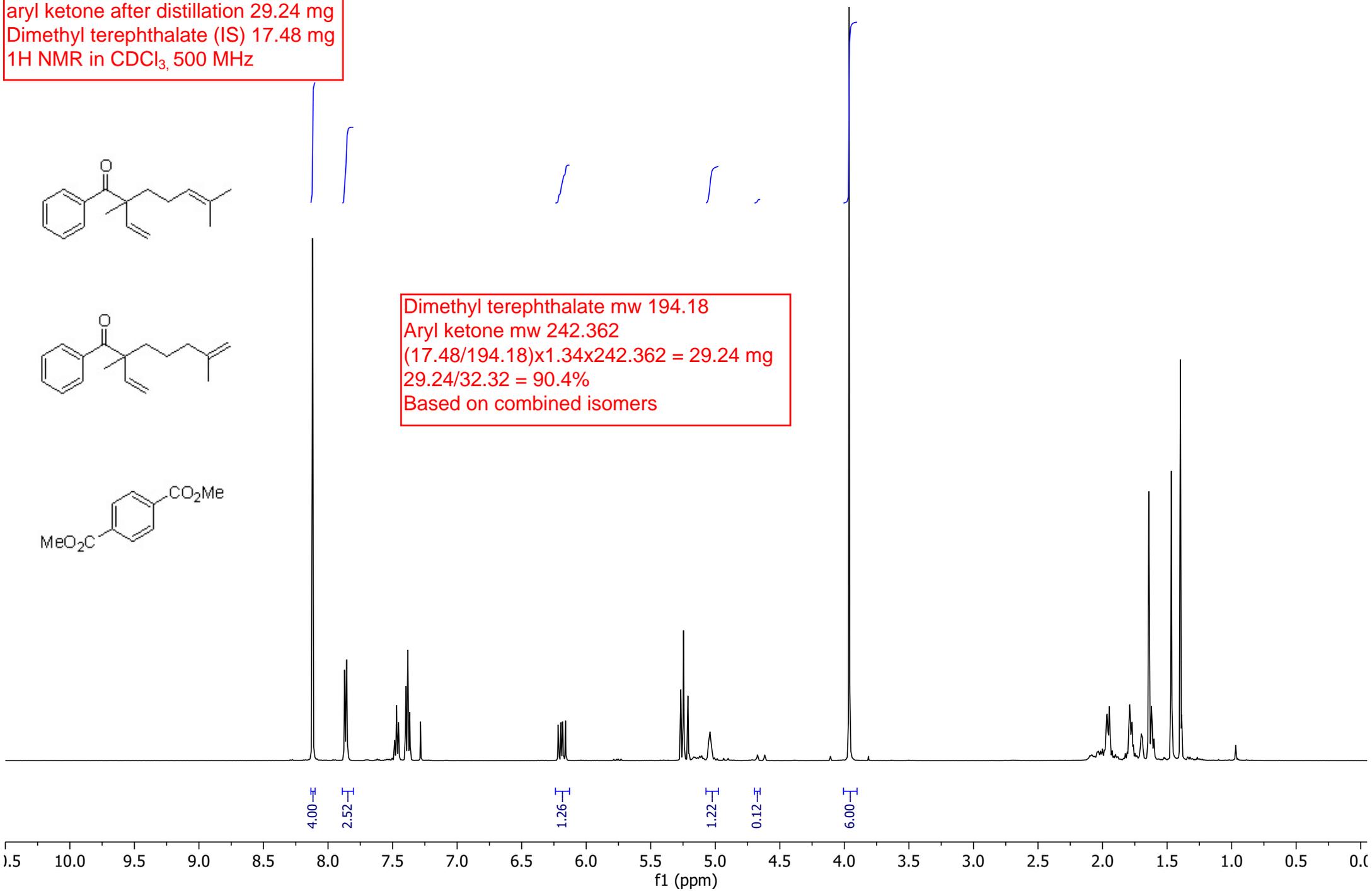
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4.67  
4.62

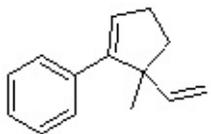
3.96

1.96  
1.95  
1.79  
1.64  
1.62  
1.47  
1.40  
1.39

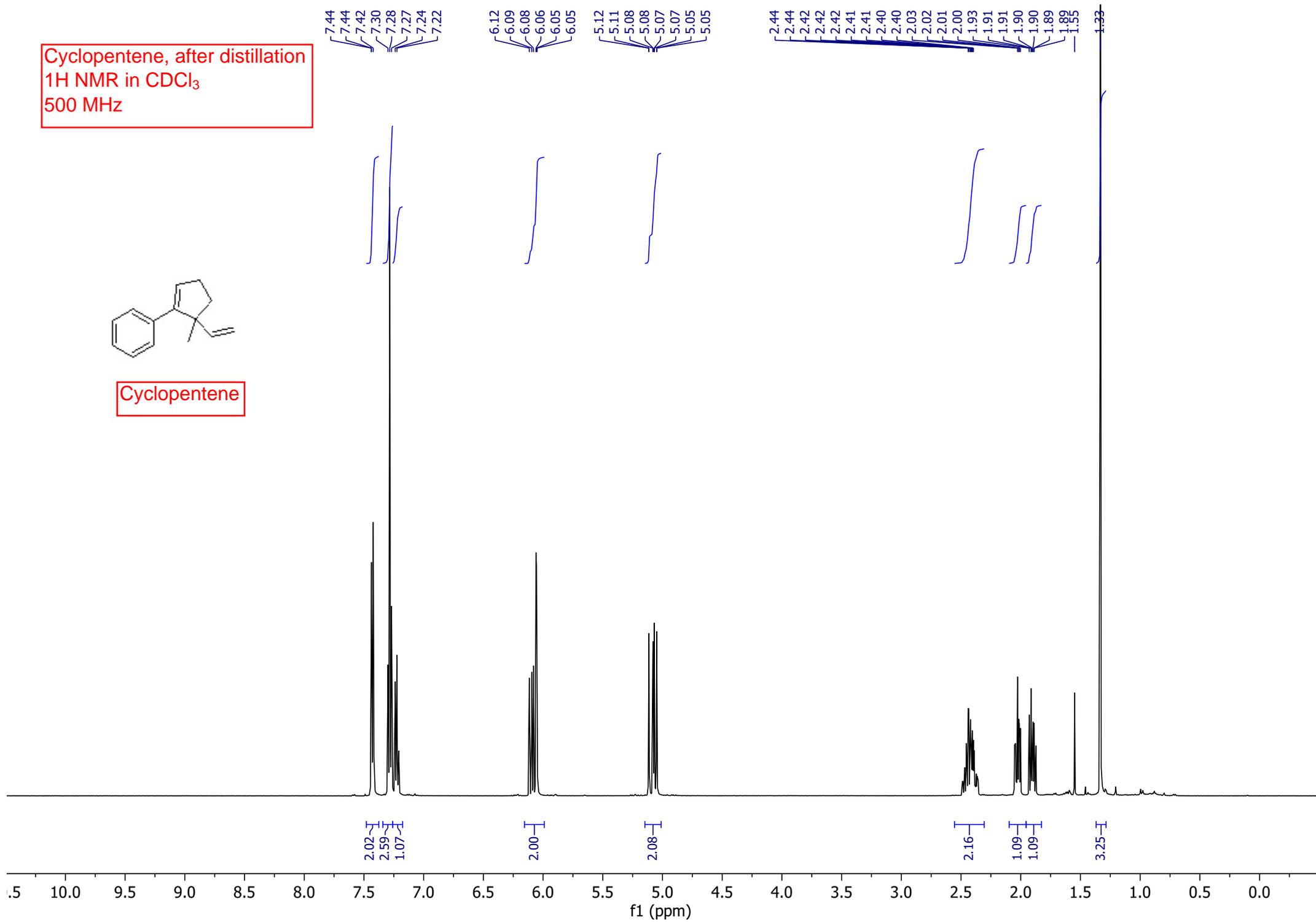
Dimethyl terephthalate mw 194.18  
Aryl ketone mw 242.362  
 $(17.48/194.18) \times 1.34 \times 242.362 = 29.24$  mg  
 $29.24/32.32 = 90.4\%$   
Based on combined isomers



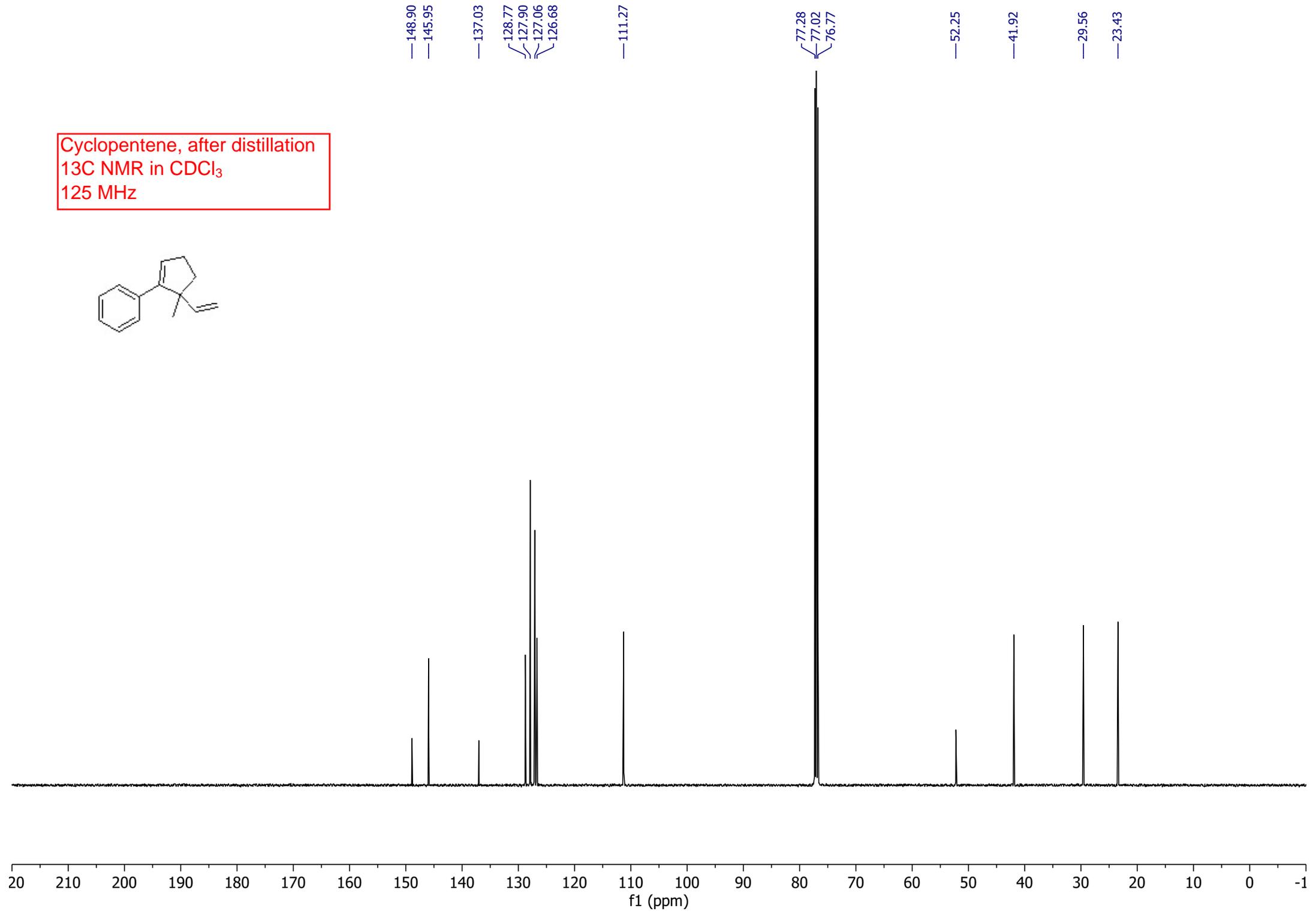
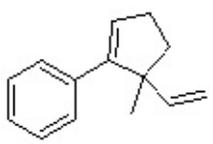
Cyclopentene, after distillation  
1H NMR in CDCl<sub>3</sub>  
500 MHz



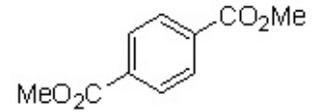
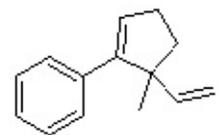
Cyclopentene



Cyclopentene, after distillation  
13C NMR in CDCl<sub>3</sub>  
125 MHz



Cyclopentene (run #1) 33.89 mg  
 Dimethyl terephthalate (IS) 16.63 mg  
 1H NMR in CDCl<sub>3</sub>, 500 MHz



DMT mw 194.18  
 Cyclopentene mw 184.282  
 $(16.63/194.18) \times 4.18 \times 184.282 / 2 = 32.99 \text{ mg}$   
 $32.99 / 33.89 = 97.3 \text{ wt\%}$   
 $(16.63/194.18) \times 4.17 \times 184.282 / 2 = 32.91 \text{ mg}$   
 $32.91 / 33.89 = 97.1 \text{ wt\%}$   
 Avg = 97.2 wt%

