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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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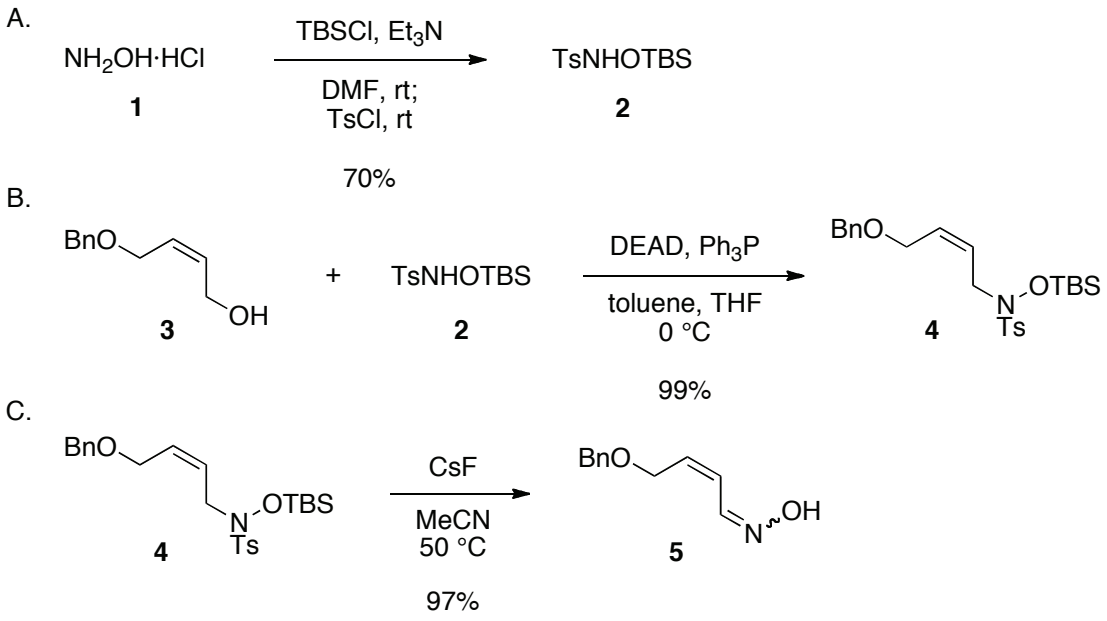
Efficient Synthesis of Oxime Using *O*-TBS-*N*-Tosylhydroxylamine: Preparation of (2*Z*)-4-(Benzyloxy)but-2-enal Oxime



Submitted by Katsushi Kitahara, Tatsuya Toma, Jun Shimokawa and Tohru Fukuyama.[1]
Checked by Erica Benedetti and Kay M. Brummond.

1. Procedure

A. *O-TBS-N-tosylhydroxylamine (2).* A 1-L, three-necked round-bottomed flask equipped with an internal thermometer, a 100-mL pressure-equalizing dropping funnel sealed with a rubber septum, a calcium chloride drying tube, and a 5.0-cm oval, Teflon-coated, magnetic stir bar, is charged under air with hydroxylamine hydrochloride (6.08 g, 87.5 mmol, 1.10 equiv) and *tert*-butyldimethylsilyl chloride (13.2 g, 87.5 mmol, 1.10 equiv) in 400 mL of DMF (Note 1). The clear solution is stirred (Note 2) in an ice bath for 30 min, then Et_3N (55.0 mL, 397.7 mmol, 5.00 equiv) is added dropwise from the dropping funnel over 20 min, such that the internal temperature does not exceed 10 °C (Note 3). A white precipitate is observed during the addition. The ice bath is removed and the suspension is vigorously stirred for 2 h (Note 4). The reaction mixture is cooled in an ice bath for 15 min and *p*-toluenesulfonyl chloride (15.1 g, 79.1 mmol, 1.00

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equiv) is added in three portions (5.0 g each) at 5 min intervals, such that the internal temperature does not exceed 10 °C (Note 5). The ice bath is removed and the suspension is vigorously stirred for 2 h, at which point TLC analysis shows no signs of *p*-toluenesulfonyl chloride (Notes 6 and 7). The reaction mixture is poured into *n*-hexane (300 mL), saturated aqueous NH_4Cl (250 mL) and H_2O (250 mL) in a 2-L separatory funnel. The layers are separated, and the aqueous phase is extracted with an additional *n*-hexane (2 x 250 mL). The organic solutions are combined, washed with H_2O (2 x 500 mL), 10% aqueous citric acid (2 x 500 mL), brine (250 mL) and dried over magnesium sulfate (60 g). After filtration through a cotton plug and rinsing with 50 mL of *n*-hexane, the solution is concentrated on a rotary evaporator (35 °C, 150 mmHg, water bath) to about 100 mL (Note 8). White crystals precipitate on cooling to 0 °C for 15 min and are collected by Büchner funnel (diameter 90 mm) with a medium porosity fritted disk using suction filtration. The precipitate is washed with ice-cooled *n*-hexane (50 mL) and dried under vacuum (25 °C, 10 mmHg, 2 h) to give the first crop of *O*-TBS-*N*-tosylhydroxylamine (**2**) (16.4 g, 54.4 mmol, 69%). A second crop of crystals (1.24 g, 4.1 mmol, 5%) is obtained by dissolving the concentrated pale-yellow mother liquor in hot *n*-hexane (30 mL) and cooling in an ice bath for 30 min, followed by the filtration using 10 mL of ice-cooled *n*-hexane (Note 9).

B. *(Z)-N-(4-(Benzyloxy)but-2-en-1-yl)-N-((tert-butyldimethylsilyl) oxy)-4-methylbenzenesulfonamide (**4**).* An oven-dried, 1-L, three-necked round-bottomed flask is equipped with a 5.0-cm oval Teflon-coated magnetic stir bar, an internal thermometer, a 50-mL pressure-equalizing dropping funnel sealed with a rubber septum, and fitted with nitrogen gas inlet adaptor. The flask is evacuated and refilled with nitrogen three times, then charged with *O*-TBS-*N*-tosylhydroxylamine (**2**) (16.1 g, 53.4 mmol, 1.00 equiv), triphenylphosphine (18.2 g, 69.3 mmol, 1.30 equiv), toluene (135 mL) and THF (45 mL) (Note 10). *cis*-4-Benzyloxy-2-buten-1-ol (**3**) (9.5 mL, 56.1 mmol, 1.05 equiv) (Note 11) is added and the solution is stirred (Note 12) in an ice bath for 10 min. Diethyl azodicarboxylate (2.2 M in toluene, 29.1 mL, 64.1 mmol, 1.20 equiv) (Note 13) is added dropwise from dropping funnel over 20 min, such that the internal temperature does not exceed 10 °C. The solution becomes cloudy during the addition (Note 14). The mixture is stirred at 0 °C for 30 min, then H_2O (500 μL) is added and the solution is evaporated (34 °C, 70 mmHg). *n*-Hexane (300 mL) is added at ambient temperature and the white solid that precipitates is

removed in a sintered-glass Büchner funnel (diameter 80 mm) using suction filtration and washed with *n*-hexane (500 mL, ambient temperature), and the filtrate is evaporated (34 °C, 70 mmHg) to give 30.5 g of the crude product as a clear, yellow oil. This material is purified by silica gel column chromatography (elution with ethyl acetate/hexane) (Notes 15 and 16). The combined eluents are concentrated by rotary evaporation (40 °C, 27 mmHg) and then dried at 25 °C (3 mmHg) for 2 h to furnish 21.7 g (47.0 mmol, 88%) of (*Z*)-*N*-(4-(benzyloxy)but-2-en-1-yl)-*N*-((*tert*-butyldimethylsilyl)-oxy)-4- methylbenzenesulfonamide (**4**) as a clear colorless oil (Note 17).

C. *(2Z)-4-(Benzyloxy)but-2-enal oxime (**5**).* An oven-dried, 500-mL, three-necked round-bottomed flask is equipped with a 3.0-cm oval Teflon-coated magnetic stir bar, a rubber septum, an internal thermometer, and fitted with nitrogen gas inlet adaptor. The flask is evacuated and refilled with nitrogen three times, then charged with (*Z*)-*N*-(4-(benzyloxy)but-2-en-1-yl)-*N*-((*tert*-butyldimethylsilyl)oxy)-4-methylbenzenesulfonamide (**4**) (21.7 g, 47.0 mmol, 1.00 equiv) and 130 mL of acetonitrile (Note 18). The septum is removed temporarily and cesium fluoride (9.29 g, 61.1 mmol, 1.30 equiv) (Note 19) is added in one portion. The resulting white suspension is stirred (Note 20) and heated in an oil bath for 2 h, so that the internal temperature is maintained at 50 °C (Note 21). The reaction mixture is cooled in an ice bath for 20 min and saturated aqueous sodium bicarbonate solution (250 mL) is added and the reaction mixture is poured into EtOAc (250 mL) in a 2-L separatory funnel. The layers are separated, and the aqueous phase is extracted with additional EtOAc (2 x 150 mL). The organic solutions are combined, washed with 150 mL of brine, and dried over sodium sulfate (30 g). Filtration through a cotton plug, rinsing with 70 mL of EtOAc and concentration on the rotary evaporator (35 °C, 42 mmHg, water bath) provides the crude oil (11.4 g). This material is purified by silica gel column chromatography (Note 22). The combined eluents are concentrated by rotary evaporation (37 °C, 34 mmHg) and then dried at 25 °C (3 mmHg) for 2 h to furnish 8.49 g (44.4 mmol, 94%) of (2*Z*)-4-(benzyloxy)but-2-enal oxime (**5**) as a clear colorless oil (Note 23).

2. Notes

1. The submitters purchased hydroxylamine hydrochloride (98.0%) from Kanto Chemical Co., Inc., reagent grade DMF (>99.0%) from Wako Pure Chemical Industries, Ltd. and *tert*-butyldimethylsilyl chloride (>98.0%)

from Tokyo Chemical Industry Co., Ltd. and used as received. The checkers purchased hydroxylamine hydrochloride (99.0%) from Sigma-Aldrich Chemical Company Inc., anhydrous DMF (99.8%) from Sigma-Aldrich Chemical Company Inc. and *tert*-butyldimethylsilyl chloride (98.0%) from Acros Organics Co. and used as received.

2. The submitters reported a stirring speed of 900 rpm.

3. The submitters purchased triethylamine (>99.0%) from Kanto Chemical Co., Inc. and used as received. The checkers purchased triethylamine (>99.0%) from Sigma-Aldrich Chemical Company Inc. and used as received.

4. The submitters reported it is necessary to stir the mixture vigorously for at least 50 minutes after the internal temperature reached ambient temperature for reproducible results. The checkers confirmed that the consumption of the starting material **1** could not be monitored by TLC analysis.

5. The submitters purchased *p*-toluenesulfonyl chloride (>99.0%) from Tokyo Chemical Industry Co., Ltd. and used as received. The checkers purchased *p*-toluenesulfonyl chloride (>99.0%) from Sigma-Aldrich Chemical Company Inc., and used as received.

6. The submitters reported it is necessary to stir the mixture for at least 30 minutes after the internal temperature reached ambient temperature for reproducible results.

7. The consumption of *p*-toluenesulfonyl chloride was monitored by TLC analysis on Whatman Ltd. silica gel 60 F_{254} plates (0.25 mm, aluminum-backed, visualized with 254 nm UV lamp and stained with phosphomolybdic acid) using 25% ethyl acetate in *n*-hexane as an eluant. *p*-Toluenesulfonyl chloride had R_f = 0.67 (UV active, black after staining) and *O*-TBS-*N*-tosylhydroxylamine (**2**) had R_f = 0.54 (UV active, black after staining).

8. The submitters observed the development of crystals during evaporation. The checkers did not observe crystals during evaporation.

9. The submitters observed that the appearance of the crystals depends on the workup procedure. Without washing with aqueous citric acid solution, low-density wooly crystals were obtained. Reagent **2** can be stored without decomposition over 3 months in the refrigerator (–30 °C) under argon atmosphere. A yield of 65% was obtained when the reaction was performed at half scale. The product displayed the following physicochemical properties: mp 122–123 °C; IR (neat, cm^{-1}) 3213, 2957,

2931, 2889, 2859, 1598, 1169; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 0.18 (s, 6 H), 0.87 (s, 9 H), 2.45 (s, 3 H), 6.43 (s, 1 H), 7.34 (d, J = 8.1 Hz, 2 H), 7.80 (d, J = 8.2 Hz, 2 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: –5.3, 18.0, 21.8, 26.0, 129.0, 129.6, 133.3, 144.8; HRMS calcd for $C_{13}H_{23}NO_3SSiNa$ ($[M + Na]^+$) 324.1066, found 324.1075; Anal. Calcd. for $C_{13}H_{23}NO_3SSi$: C, 51.79; H, 7.69; N, 4.65; Found: C, 51.65; H, 7.69; N, 4.66 (Submitters-in house analysis); Found C, 51.26; H, 7.29; N, 4.86 (Checkers-sent out for analysis).

10. The submitters purchased triphenylphosphine (>95.0%) from Tokyo Chemical Industry Co., Ltd., dehydrated toluene (>99.5%) and dehydrated THF (>99.5%) from Kanto Chemical Co., Inc. and used the material as received. The checkers purchased triphenylphosphine (99.0%), anhydrous toluene (99.8%) and anhydrous THF (>99.9%) from Sigma-Aldrich Chemical Company Inc. and used as received.

11. The submitters and the checkers purchased *cis*-4-benzyloxy-2-buten-1-ol (**3**) (>95%) from Sigma-Aldrich Chemical Company Inc. and used the material as received.

12. The submitters reported a stirring speed of 540 rpm.

13. The submitters purchased diethyl azodicarboxylate (2.2 M in toluene, 40%) from Tokyo Chemical Industry Co., Ltd. and used as received. The checkers purchased diethyl azodicarboxylate (2.2 M in toluene, 40%) from Chem-Impex International, Inc. and used as received.

14. The consumption of the starting material was monitored by TLC analysis on Whatman Ltd. silica gel 60 F_{254} plates (0.25 mm, aluminum-backed, visualized with 254 nm UV lamp and stained with phosphomolybdic acid) using 25% ethyl acetate in *n*-hexane as an eluant. Ph_3P had R_f = 0.79 (UV active, white after staining), **4** had R_f = 0.68 (UV active, black after staining), **2** had R_f = 0.55 (UV active, black after staining) and **3** had R_f = 0.18 (UV weakly active, black after staining).

15. Silica gel (pH range: 6.5–7.5) was purchased from Sorbent Technologies, Inc. (40-63 μm).

16. The crude material is dissolved in *n*-hexane (10 mL) and then is charged onto a column (diameter = 6.5 cm, height = 5.5 cm) of 170 g (300 mL) of silica gel. The column was eluted with *n*-hexane (1500 mL) to elute Ph_3P, *n*-hexane/EtOAc (20:1) (300 mL) and *n*-hexane/EtOAc (10:1) (1000 mL) and 30-mL fractions were collected. Fractions 52-87 were collected.

17. A yield of 75% was obtained when the reaction was performed at half scale. The product displayed the following physicochemical properties:

IR (neat, cm^{-1}) 2954, 2930, 2887, 2858, 1597, 1360, 1170; ^1H NMR ($CDCl_3$, 400 MHz) δ: 0.26 (s, 6 H), 0.90 (s, 9 H), 2.45 (s, 3 H), 3.66 (d, *J* = 6.1 Hz, 2 H), 3.98 (d, *J* = 6.1 Hz, 2 H), 4.45 (s, 2 H), 5.58 (dt, *J* = 12.3, 6.2 Hz, 1 H), 5.70 (dt, *J* = 12.3, 6.2 Hz, 1 H), 7.25–7.35 (m, 7 H), 7.70 (d, *J* = 8.2 Hz, 2 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 4.1, 18.2, 21.8, 26.1, 52.9, 65.8, 72.5, 126.4, 127.76, 127.80, 128.5, 129.4, 130.0, 130.20, 130.26, 138.1, 144.7; HRMS calcd for $C_{24}H_{35}NO_4SSiNa$ ($[M + Na]^+$) 484.1954, found 484.1978. Anal. Calcd. for $C_{24}H_{35}NO_4SSi$: C, 62.44; H, 7.64; N, 3.03; Found: C, 62.36; H, 7.53; N, 3.13.

18. The submitters purchased dehydrated acetonitrile (99.5%) from Kanto Chemical Co., Inc. and used as received. The checkers purchased anhydrous acetonitrile (99.8%) from Sigma-Aldrich Chemical Company Inc. and used as received.

19. The submitters purchased cesium fluoride (97%) from Wako Pure Chemical Industries, Ltd. and used as received. The checkers purchased cesium fluoride (99.0%) from Sigma-Aldrich Chemical Company Inc. and used as received.

20. The submitters reported a stirring speed of 540 rpm.

21. The consumption of the starting material was monitored by TLC on Whatman Ltd. silica gel 60 F_{254} plates (0.25 mm, aluminum-backed, visualized with 254 nm UV lamp and stained with cerium phosphomolybdic acid) using 25% ethyl acetate in *n*-hexane as an eluant. Compound **5** had R_f = 0.33 and 0.23 (*E-Z* mixture, UV active, black after staining).

22. The crude material is dissolved in 10 mL of *n*-hexane/EtOAc (6:1) and then is charged onto a column (diameter = 6.5 cm, height = 3.7 cm) of 86 g (200 mL) of silica gel. The column was eluted with *n*-hexane/EtOAc (6:1) and 30-mL fractions were collected. Fractions 24-92 were collected.

23. A yield of 95% was obtained when the reaction was performed at half scale. The product was isolated as a 1.4:1 *E-Z* isomeric mixture and displayed the following physicochemical properties: IR (neat, cm^{-1}) 3286, 3063, 3032, 2859, 1454; ^1H NMR ($CDCl_3$, 400 MHz) δ: 4.22 (dd, *J* = 6.4, 1.8 Hz, 2 H *major*), 4.24 (dd, *J* = 6.4, 1.8 Hz, 2 H *minor*), 4.55 (s, 3 H *major*), 4.56 (s, 3 H *minor*), 5.99–6.10 (m, 1 H *minor* and 1 H *major*), 6.23 (t, *J* = 11.3 Hz, 1 H *major*), 6.83 (td, *J* = 10.0, 1.4 Hz, 1 H *minor*), 7.29–7.38 (m, 5 H *major* and 5 H *minor*), 7.46 (d, *J* = 10.4 Hz, 1 H *minor*), 8.11 (d, *J* = 10.4 Hz, 1 H *major*); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 65.98, 65.99, 72.63, 72.70, 118.1, 124.2, 128.1, 128.6, 134.5, 136.3, 137.6, 137.7, 144.1, 147.5; HRMS calcd for $C_{11}H_{14}NO_2$ ($[M + H]^+$) 192.1025, found 192.1013. Anal.

Calcd. for $C_{11}H_{13}NO_2$: C, 62.09; H, 6.85; N, 7.32; Found: C, 61.68; H, 6.39; N, 7.54.

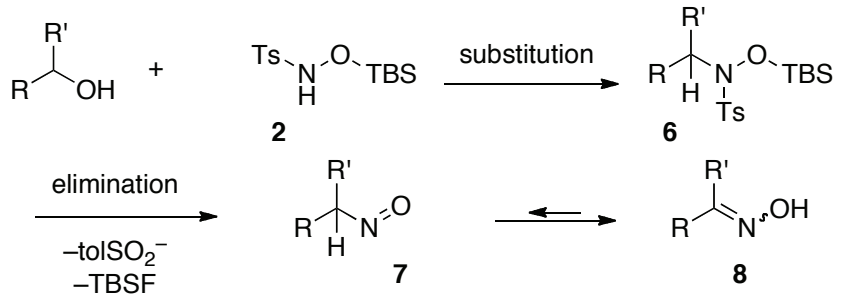
Safety and Waste Disposal Information

All hazardous materials should be handled and disposed of in accordance with "Prudent Practices in the Laboratory"; National Academies Press; Washington, DC, 2011.

3. Discussion

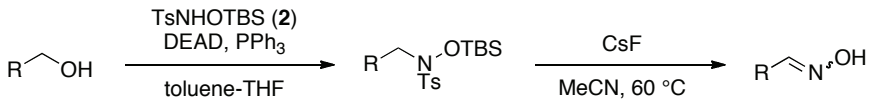
The oxime functionality is most often prepared by the reactions between carbonyl compounds and hydroxylamine. This sometimes suffers from a problem if an aldehyde or a ketone is not stable enough or possesses low reactivity for the transformation. This drawback could be circumvented if the oximes are prepared without going through aldehydes or ketones. We envisaged that the elimination of a sulfonyl group attached to a hydroxylamine moiety in **6** would furnish access to an alkyl nitroso intermediate **7**, which would in turn tautomerize quickly to give oxime **8** (Scheme 1). This transformation bypasses the carbonyl intermediates and thus would not be troubled by the problems concerning the reactivity of a carbonyl intermediate. The procedure presented here is a convenient method for the synthesis of (2*Z*)-4-(benzyloxy)but-2-enal oxime (**5**) from the allylic alcohol **3** and *O*-TBS-*N*-tosylhydroxylamine (**2**)[3,4] minimal isomerization from *Z* to *E* within the unsaturated system.[4] The procedure utilizing a Mitsunobu reaction and subsequent treatment with cesium fluoride was found to be applicable to the preparation of various oximes shown in Table 1.

Scheme 1. Proposed mechanism.



Bromides, tosylates, and mesylates can also be used as precursors in the one-pot transformation to oximes by means of an S_N2 reaction and subsequent elimination (Scheme 2).

Table 1. Substrate Scope.



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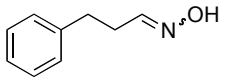
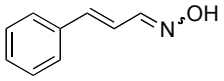
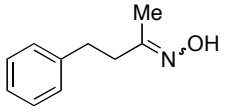
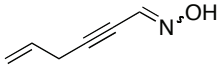
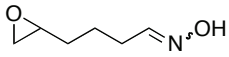
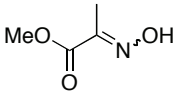
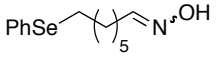
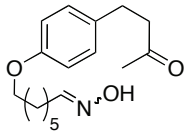
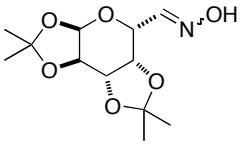
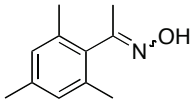
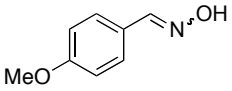
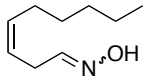
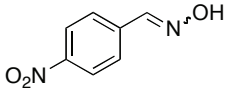
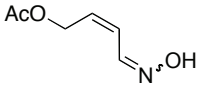
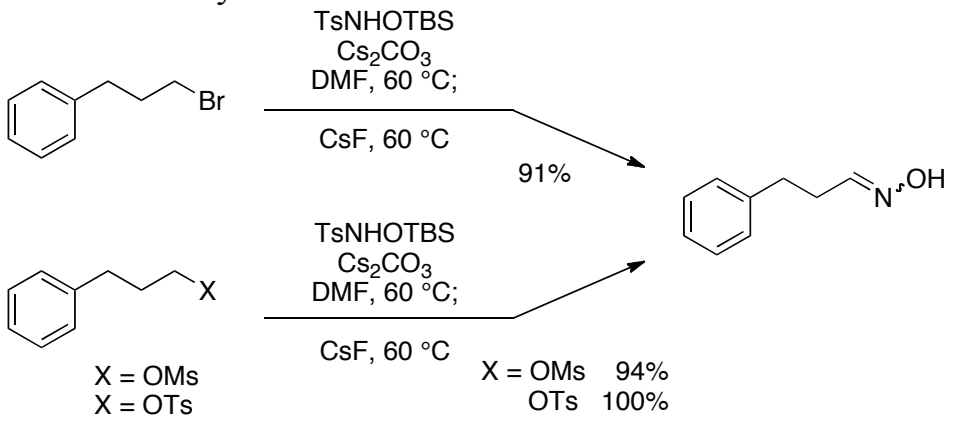
1		99 / 99	8		99 / 94
2		91 / 92	9		100 / 92[e]
3		100 / 82	10		95 / 95[e]
4		97 / 98	11		85 / 99
5		84[c] / 90	12		99[f] / 94
6		91 / 99	13		99 / 89
7		97[d] / 91	14		93 / 98

[a] Standard conditions for the Mitsunobu reaction: 1.1 equiv of alcohol, 1.0 equiv of TsNHOTBS, 1.5 equiv of DEAD, 2.0 equiv of Ph_3P, toluene-THF (3:1, 0.2 M), 0 °C. Standard conditions for oxime formation: 2.0 equiv of CsF, MeCN (0.1 M), 60 °C. [b] Isolated yields for Mitsunobu reaction / oxime formation reaction. [c] Mitsunobu reaction was conducted with 2.5 equiv of DEAD and 3.0 equiv of Ph_3P. [d] A minimal amount (1.05 equiv) of DEAD was used because of the instability of the product with the reagent. [e] 2.0 equiv of AcOH was added for buffering the basicity. [f] Mitsunobu reaction was conducted with 1.5 equiv of alcohol and 2.0 equiv of DEAD at 40 °C.

Scheme 2. From Alkyl Bromide and Sulfonate.



1. Graduate School of Pharmaceutical Sciences, University of Tokyo, Hongo 7-3-1, Bunkyo-ku, Tokyo 113-0033, Japan; Email: fukuyama@mol.f.u-tokyo.ac.jp. We are grateful for the Grant-in-Aid (15109001 and 18890048) from the Ministry of Education, Culture, Sports, Science, and Technology of Japan.

2. Kitahara, K.; Toma, T.; Shimokawa, J.; Fukuyama, T. *Org. Lett.* **2008**, *10*, 2259.

3. For selected applications of this method, see; (a) Rai, G.; Thomas, C. J.; Leister, W.; Maloney, D. J. *Tetrahedron Lett.* **2009**, *50*, 1710; (b) Singh, M. K.; Lakshman, M. K. *J. Org. Chem.* **2009**, *74*, 3079; (c) Su, D.-Y.; Wang, X.-Y.; Shao, C.-W.; Xu, J.-M.; Zhu, R.; Hu, Y.-F. *J. Org. Chem.* **2011**, *76*, 188.

4. Enev, V. S.; Drescher, M.; Kählig, H.; Mulzer, J. *Synlett* **2005**, 2227. According to the authors, this was the first example of a stereoselective synthesis of a *cis*-α,β-unsaturated oxime.

Appendix
Chemical Abstracts Nomenclature; (Registry Number)

Hydroxylamine hydrochloride: Hydroxylamine, hydrochloride (1:1); (5470-11-1)

tert-Butyldimethylsilyl chloride: Silane, chloro(1,1-dimethylethyl)dimethyl-; (18162-48-6)

Dimethylformamide: Formamide, *N*,*N*-dimethyl-; (68-12-2)

Triethylamine: Ethanamine, *N*,*N*-diethyl-; (121-44-8)

p-Toluenesulfonyl chloride: Benzenesulfonyl chloride, 4-mehyl-; (98-59-9)

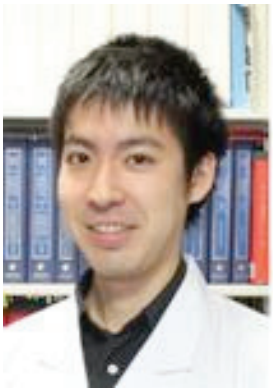
O-TBS-*N*-tosylhydroxylamine: Benzenesulfonamide, *N*-[[(1,1-dimethylethyl)dimethylsilyl]oxy]-4-methyl-; (1028432-04-3)

Ph_3P: Phosphine, triphenyl-; (603-35-0)

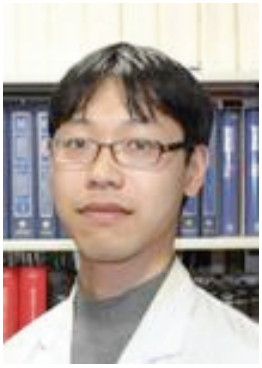
4-Benxyloxy-2-buten-1-ol: 2-Buten-1-ol, 4-(phenylmethoxy)-, (2*Z*)-; (81028-03-7)

DEAD: 1,2-Diazenedicarboxylic acid, 1,2-diethyl ester; (1972-28-7)

Cesium fluoride; (13400-13-0)



Katsushi Kitahara was born in 1984 in Saitama, Japan. He received his B.S. in 2007 from Tokyo University of Science, where he carried out his undergraduate research in the laboratories of Susumu Kobayashi. The same year he started his doctoral study at the University of Tokyo under the supervision of Professor Tohru Fukuyama. His current interest is enantioselective total synthesis of complex natural products.



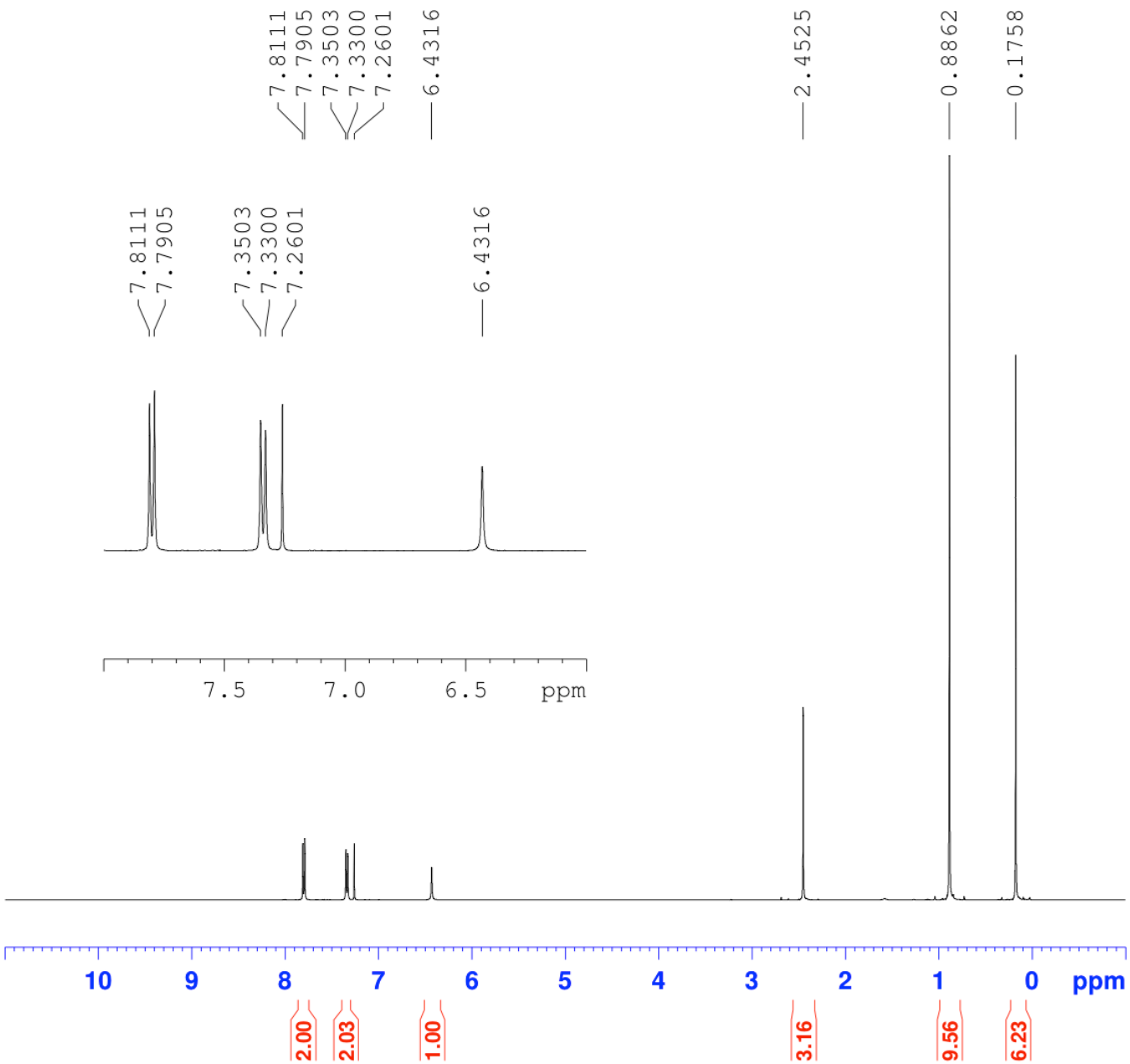
Tatsuya Toma was born in 1984 in Saitama, Japan. He graduated in 2007 and received his M. S. degree in 2009 from the University of Tokyo. The same year he started his Ph. D. study under the supervision of Professor Tohru Fukuyama. His current interest is enantioselective total synthesis of complex natural products.

Jun Shimokawa was born in 1980 in Tokyo. He performed his Ph.D studies under the direction of Professor Tohru Fukuyama at the University of Tokyo where he conducted the research on total syntheses of complex natural products. In 2006, he was appointed as Assistant Professor in the same group. His research efforts focus on the development of novel synthetic methodology and applications to the synthesis of complex molecule.

Tohru Fukuyama received his Ph.D. in 1977 from Harvard University with Yoshito Kishi. He remained in Kishi's group as a postdoctoral fellow until 1978 when he was appointed as Assistant Professor of Chemistry at Rice University. After seventeen years on the faculty at Rice, he returned to his home country and joined the faculty of the University of Tokyo in 1995, where he is currently Professor of Pharmaceutical Sciences. He has primarily been involved in the total synthesis of complex natural products of biological and medicinal importance. He often chooses target molecules that require development of new concepts in synthetic design and/or new methodology for their total synthesis.

Erica Benedetti was born in 1984 in Como, Italy. She performed her PhD studies at the Insubria University of Como (advisors: Dr. Andrea Penoni and Pr. Giovanni Palmisano), in co-tutorship with the University Pierre et Marie Curie of Paris (advisors: Dr Jean-Philippe Goddard, Pr. Louis Fensterbank and Pr. Max Malacria), where she conducted researches on transition-metal catalyzed cycloisomerization reactions. She is now a Post-doc associate at the University of Pittsburgh, under the direction of Professor Kay M. Brummond. Her current research involves the synthesis of new fluorescent compounds.

EB-006 1H NMR



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FIDRES	0.125483 Hz
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RG	80.6
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DE	6.50 usec
TE	297.6 K
D1	1.00000000 sec
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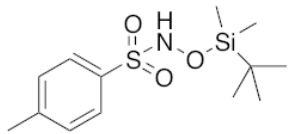
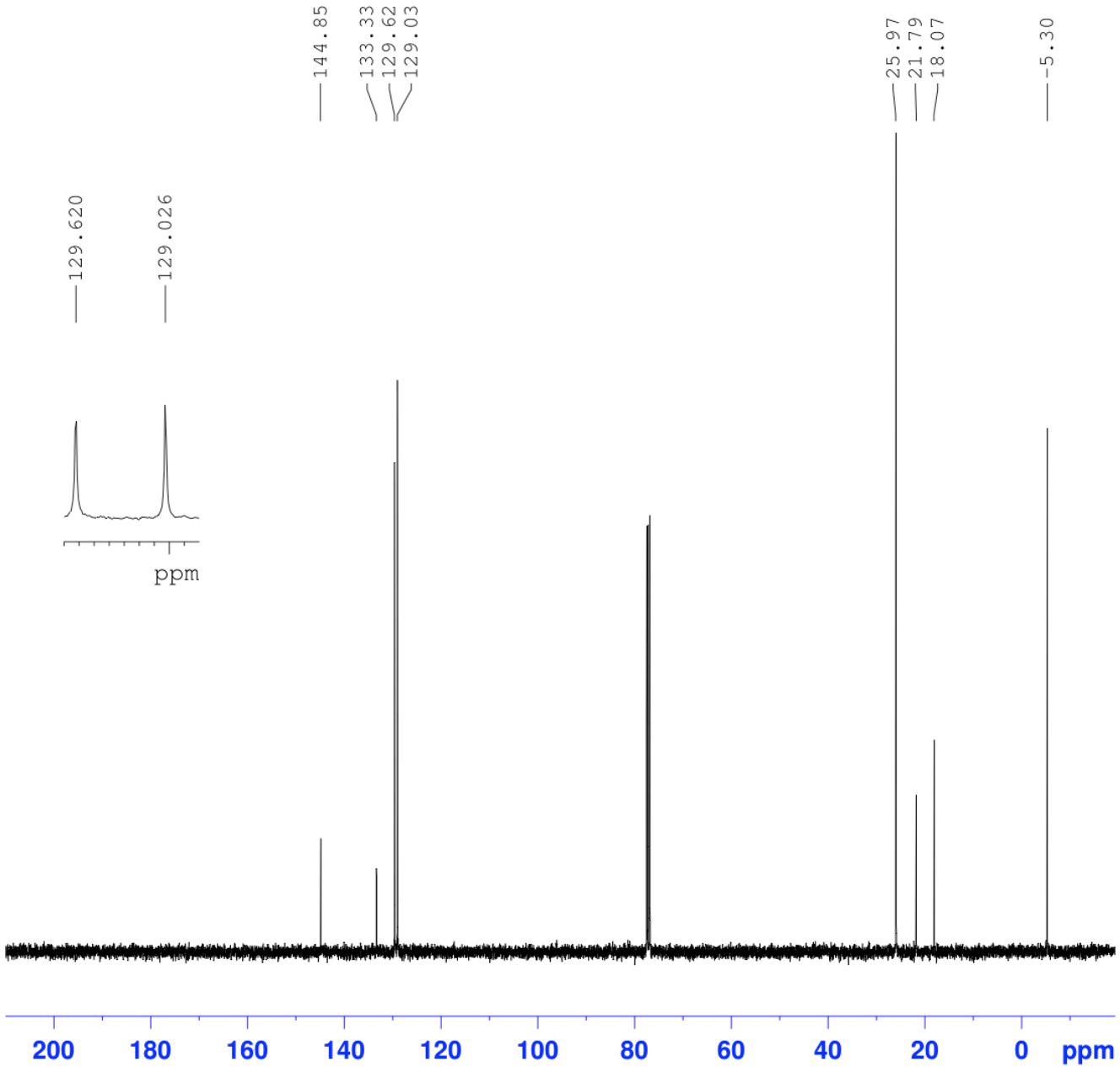


O-TBS-N-tosylhydroxylamine

EB-006 13C NMR



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FIDRES	0.366798 Hz
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TE	299.8 K
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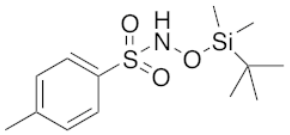
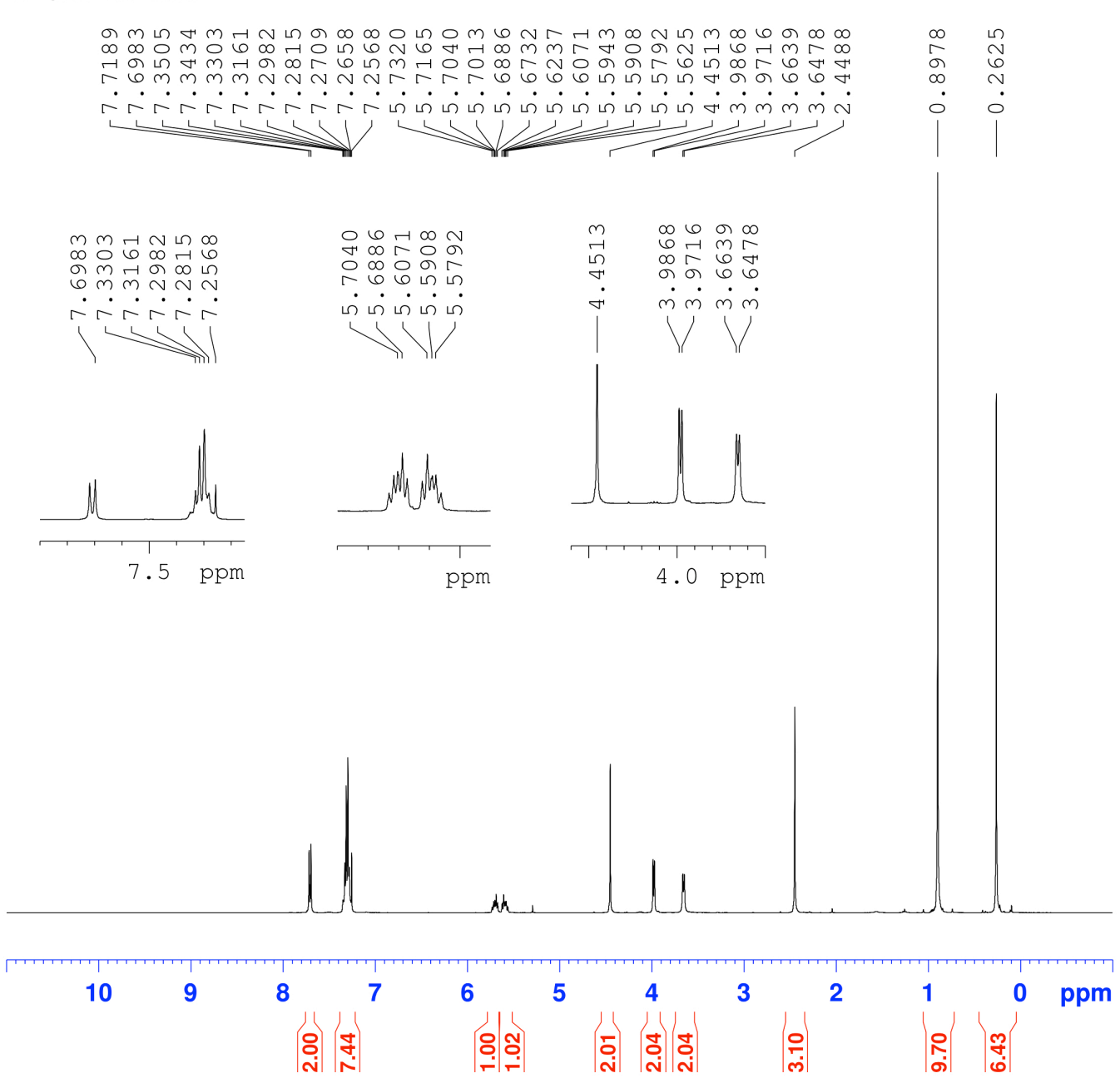


O-TBS-N-tosylhydroxylamine

EB-011-1H NMR



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FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	64
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DE	6.50 usec
TE	296.4 K
D1	1.00000000 sec

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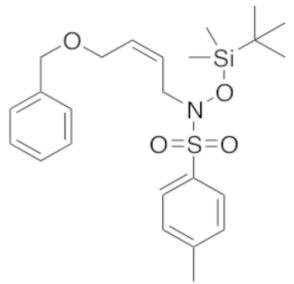
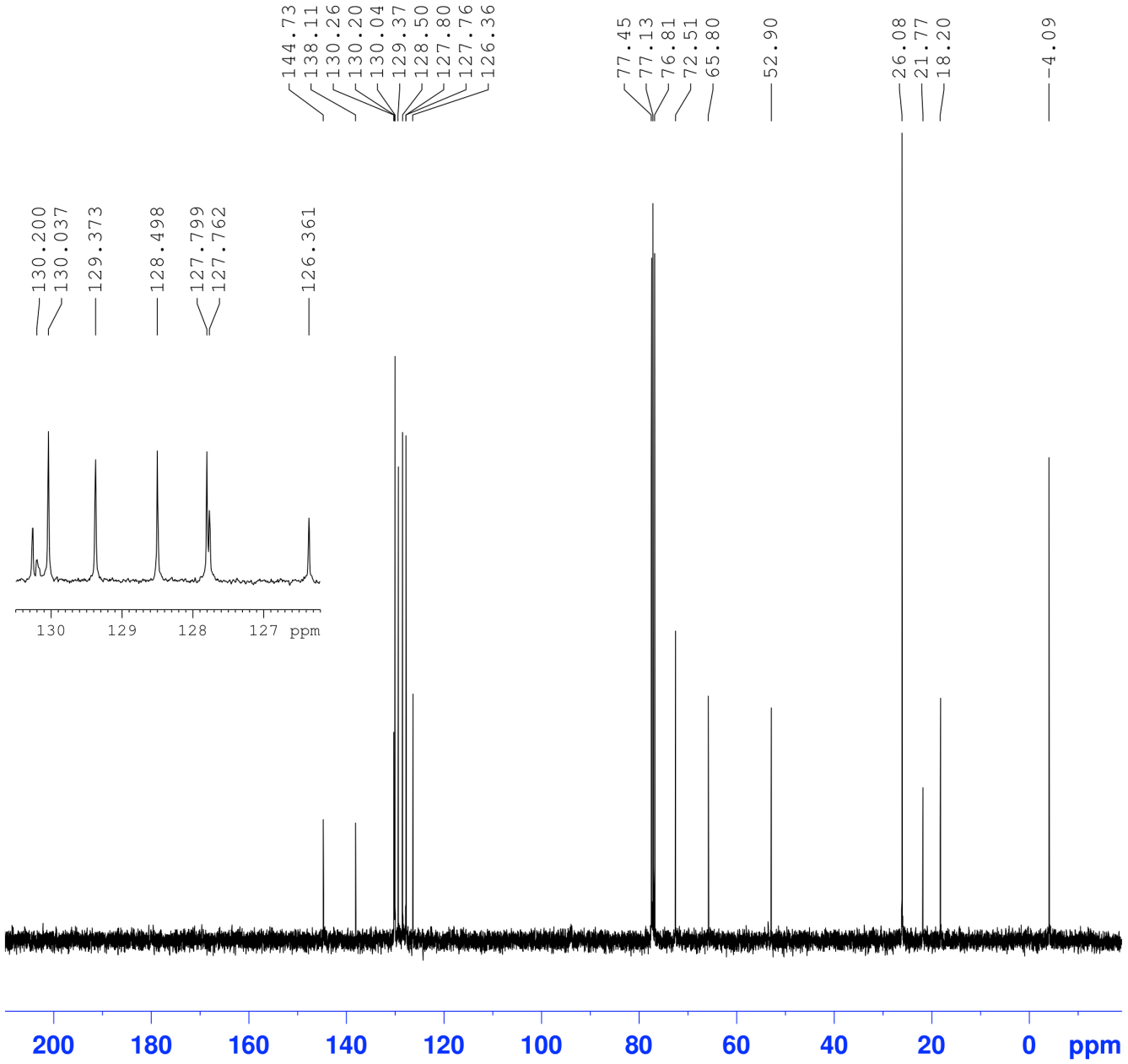


(*Z*)-*N*-(4-(benzyloxy)but-2-en-1-yl)-*N*-((tert-butyldimethylsilyl)oxy)-4-methylbenzenesulfonamide

EB-011 13C NMR



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FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	80.6
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TE	297.4 K
D1	2.00000000 sec
D11	0.03000000 sec
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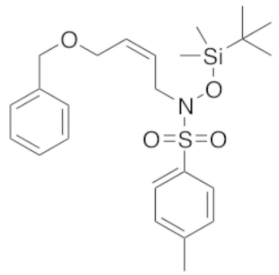
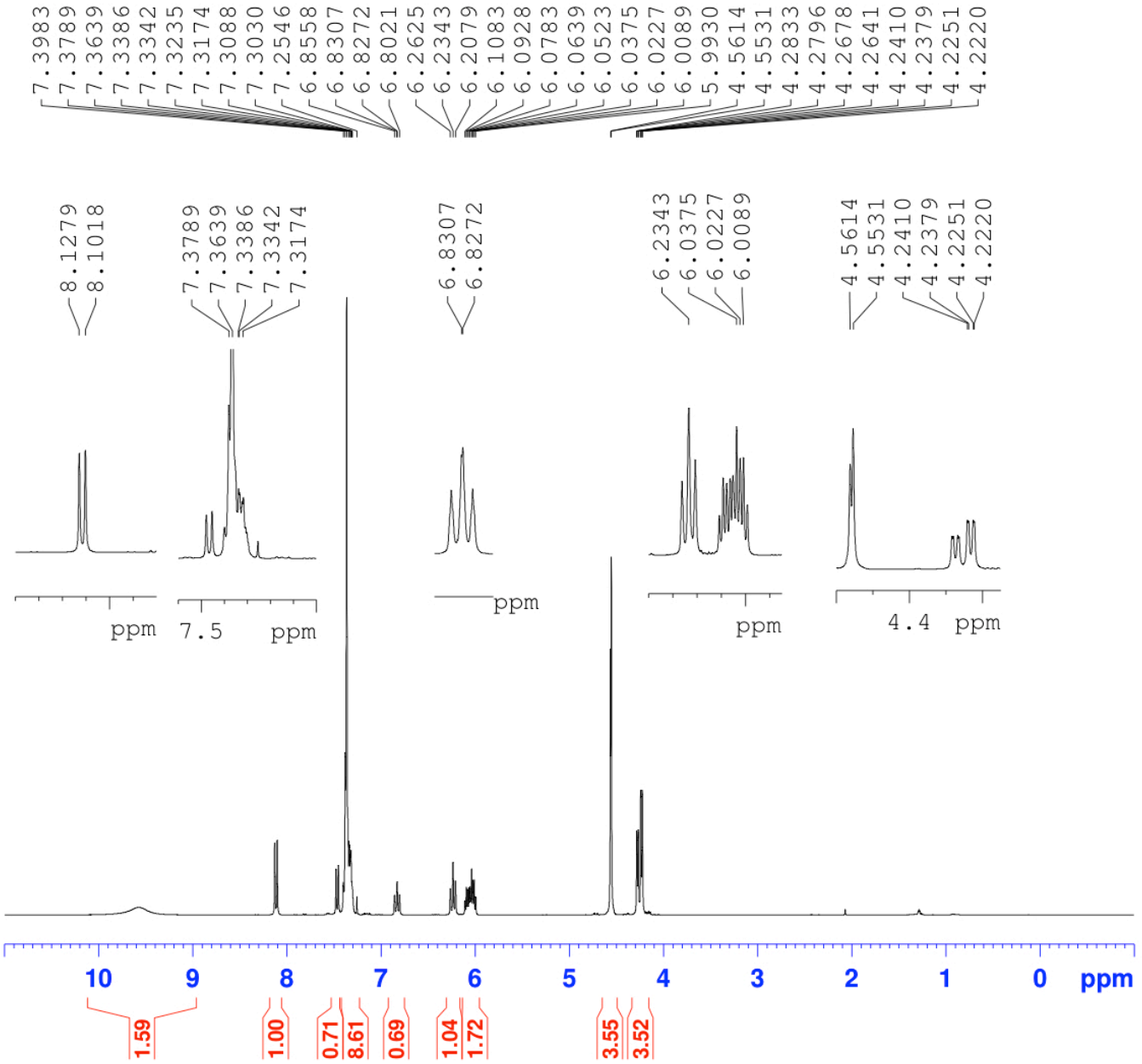
PC	1.40



(*Z*)-*N*-(4-(benzyloxy)but-2-en-1-yl)-*N*-((tert-butyldimethylsilyl)oxy)-4-methylbenzenesulfonamide

EB-021-1H NMR



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FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	18
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DE	6.50 usec
TE	295.5 K
D1	1.00000000 sec
======== CHANNEL f1 ========	
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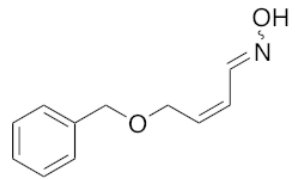
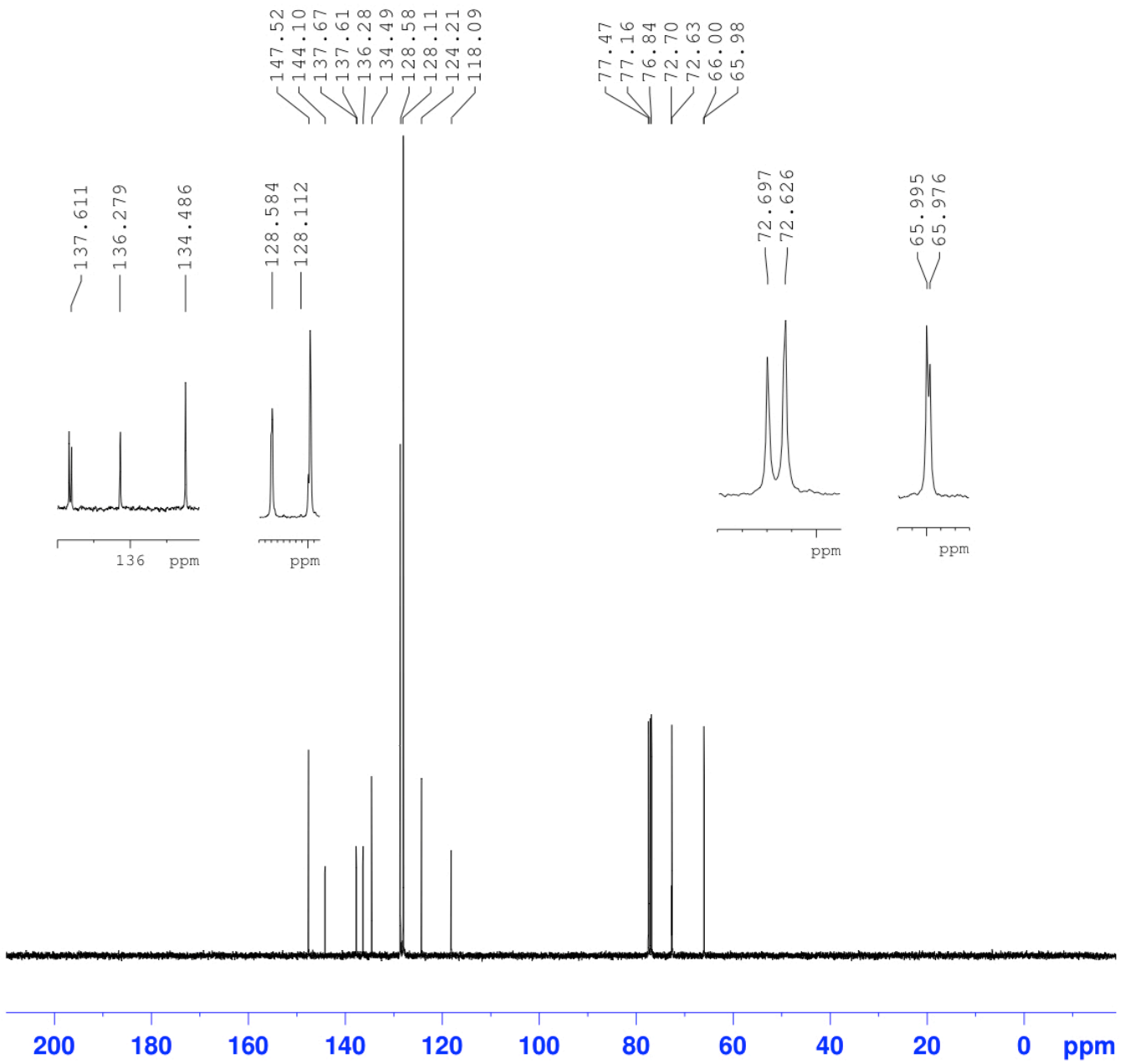


(2*Z*)-4-(benzyloxy)but-2-enal oxime

EB-021 13C NMR



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FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	296.1 K
D1	2.00000000 sec
D11	0.03000000 sec

======== CHANNEL f1 ========	
NUC1	13C
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SI	32768
SF	100.6127667 MHz
WDW	EM
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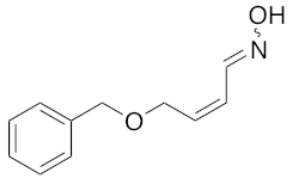
LB	1.00 Hz
GB	0
PC	1.40



(2*Z*)-4-(benzyloxy)but-2-enal oxime