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of Reliable Methods
for the Preparation
of Organic Compounds

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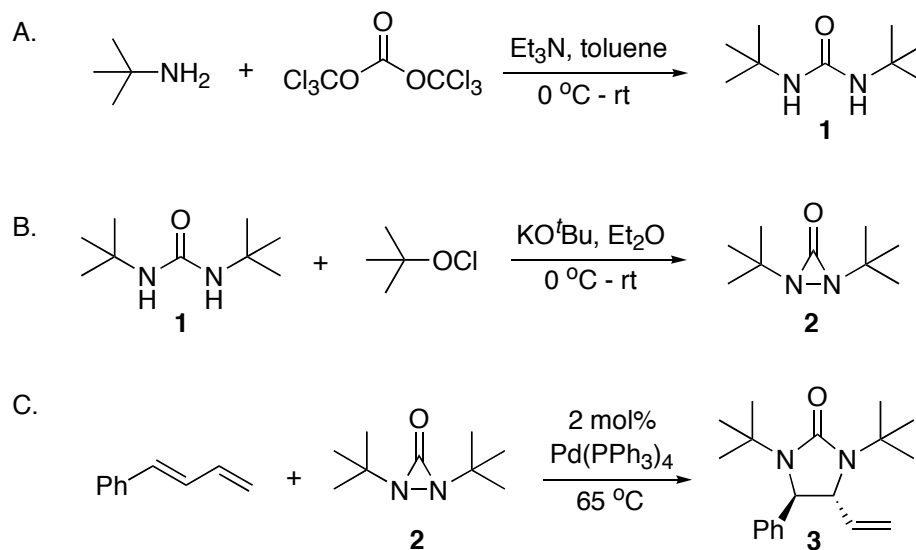
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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions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

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Pd(0)-CATALYZED DIAMINATION OF *TRANS*-1-PHENYL-1,3-BUTADIENE WITH DI-*TERT*-BUTYLDIAZIRIDINONE AS NITROGEN SOURCE



Submitted by Haifeng Du, Baoguo Zhao, and Yian Shi.*¹
 Checked by Eric E. Buck and Peter Wipf.²

1. Procedure

A. Di-tert-butylurea (1). A 5-L Erlenmeyer flask equipped with a mechanical stirrer and an internal thermometer is charged with *tert*-butylamine (85.2 mL, 791 mmol, 6.6 equiv), triethylamine (127 mL, 900 mmol, 7.5 equiv), and toluene (2000 mL) (Note 1). The mixture is cooled to an internal temperature of 5 °C in an ice bath and triphosgene (36.3 g, 120 mmol, 1.0 equiv) (Note 2) is added in small portions over a period of 20 min (Note 3). The mixture is warmed to room temperature and stirred for 24 h. The reaction mixture is quenched with water (500 mL), transferred to a separatory funnel, and the aqueous phase is discarded. The organic layer is washed with water (4 x 500 mL) and the white solids are collected by suction filtration (water aspirator) (Note 4 and 5). The combined white solids are washed with water (2000 mL) (Note 6), and dried at 65 °C under vacuum (74 mmHg) for 12 h to give 49.7 g (80%) of di-*tert*-butylurea as a white solid (Notes 7 and 8).

B. Di-tert-butyl diaziridinone (2).^{3,4} A 500-mL, one-necked, round-bottomed flask equipped with a Teflon-coated magnetic stir bar (length:

3.3 cm) and a rubber septum is charged with di-*tert*-butylurea (30.0 g, 174 mmol, 1.0 equiv) and Et₂O (200 mL) (Note 9). The reaction vessel is protected from light and *tert*-butyl-hypochlorite (20.8 g, 192 mmol, 1.1 equiv) (Note 10) is added to the slurry over a period of 10 min. The resulting pale yellow solution is stirred for an additional 30 min, cooled to 5 °C in an ice bath, and treated with potassium *tert*-butoxide (25.4 g, 226 mmol, 1.3 equiv) (Note 11) in small portions over a period of 20 min (Note 12). The resulting solution is warmed to room temperature and stirred for 5 h (Note 13). The reaction mixture is diluted with hexanes (200 mL), transferred to a 2-L separatory funnel, and washed with water (3 x 150 mL). The organic phase is dried over anhydrous K₂CO₃ (30.0 g) (Note 14) by stirring over 5 h, filtered, and concentrated by rotary evaporation (25 °C, 18 mmHg) (Note 15). The crude material is purified by fractional distillation under vacuum (7 mmHg) using a vigreux column (length 10.5 cm) fitted with a short path distillation head (Note 16). The oil bath temperature is gradually increased from 24 °C to 70 °C to give 22.6 g (76%) of di-*tert*-butyldiaziridinone as a colorless liquid (Notes 17, 18, 19, and 20).

C. *trans*-1,3-Di-*tert*-butyl-4-phenyl-5-vinyl-imidazolidin-2-one (3). A 25-mL, one-necked, round-bottomed flask equipped with a Teflon-coated magnetic stir bar (length: 1.3 cm) and a rubber septum is charged with freshly distilled (*E*)-1-phenyl-1,3-butadiene (2.60 g, 20.0 mmol, 1.0 equiv) (Note 21) and tetrakis(triphenylphosphine)palladium (0.462 g, 0.400 mmol, 0.02 equiv) (Notes 22 and 23). The flask is evacuated and back-filled with argon three times. The mixture is placed in a preheated (65 °C) oil bath and di-*tert*-butyl-diaziridinone (3.75 g, 22.0 mmol, 1.1 equiv) is added over 3 h via syringe pump (Note 24). The resulting red mixture is stirred for an additional 1 h (Note 13), cooled to room temperature, and diluted with 5 mL of a hexanes/Et₂O (5:1) solution. The solution is loaded onto a wet-packed silica gel column (305 g SiO₂, diameter: 6.5 cm, height: 22 cm, pretreated with hexanes/diethyl ether, 5:1 (Note 25). The product is eluted with hexanes/diethyl ether, 5:1) to give 5.78 g (96%) of *trans*-1,3-di-*tert*-butyl-4-phenyl-5-vinyl-imidazolidin-2-one as a yellow oil (Notes 26 and 27).

2. Notes

1. *tert*-Butylamine (98%, Alfa Aesar), triethylamine (99%, Fisher Chemicals), and toluene (99.9%, Fisher Chemicals, ACS grade) were used as received.

2. Triphosgene (98%) was purchased from Alfa Aesar and used as received.
3. The addition was carried out at such a rate that an internal temperature of 15 °C was not exceeded.
4. The solids are suspended in the organic phase.
5. The filtrate was concentrated to give an additional 2.35 g of di-*tert*-butylurea.
6. It is imperative to wash the solids extensively with water to remove any residual Et₃N•HCl, which may decrease the yield for the subsequent step.
7. Working at 50% scale, the checkers obtained 23.1 g (76%).
8. The product has the following physicochemical properties: White solid, mp 245–246 °C; ¹H NMR (300 MHz, DMSO-*d*₆) δ: 1.18 (s, 18H), 5.45 (s, 2H); ¹³C NMR (75 MHz, DMSO-*d*₆) δ: 29.3, 48.8, 157.0; IR (ATR) 3349, 1633 cm⁻¹; MS (EI+) *m/z* 172 (M⁺, 26), 157 (67), 57 (100); HRMS (EI+) *m/z* calcd for C₉H₂₀N₂O 172.1576, found 172.1569; Anal. Calcd for C₉H₂₀N₂O C, 62.75; H, 11.70; N, 16.26; Found C, 63.02; H, 11.94; N, 16.16.
9. Diethyl ether (ACS grade) was purchased from Fisher Chemicals and dried by distillation over sodium/benzophenone under an argon atmosphere.
10. *tert*-Butyl hypochlorite was prepared according to a literature procedure: Teeter, H. M.; Bell, E. W. *Org. Synth.* **1952**, 32, 20.
11. Solid potassium *tert*-butoxide (97%) was purchased from Alfa Aesar and used as received.
12. Upon complete addition the reaction vessel does not need to be protected from light.
13. The progress of the reaction can be monitored by ¹H NMR analysis of aliquots taken directly from the reaction mixture.
14. Anhydrous K₂CO₃ (ACS grade) was purchased from Fisher Chemicals and used as received.
15. The *tert*-butanol generated in the reaction should be completely removed to prevent interference with the subsequent distillation.
16. The dimensions of the distillation head are as follows. Single piece construction with inlet for vacuum/inert gas, 10/18 thermometer joint on top, 14/20 joints for distillation and collection flasks, approx. 35 mm length of condenser x 65 mm height (head).
17. Di-*tert*-butyldiaziridinone is collected at 47–49 °C (7 mmHg) and stored away from light.

18. The submitters reported a bp of di-*tert*-butyldiaziridinone of 60–64 °C (7 mmHg).

19. Working at 50% scale, the checkers obtained 11.1 g (75%).

20. The product has the following physicochemical properties: bp 47–49 °C /7 mmHg; ¹H NMR (300 MHz, benzene-*d*₆) δ: 1.07 (s, 18 H); ¹³C NMR (75 MHz, benzene-*d*₆) δ: 27.2, 59.3, 159.3; IR (ATR) 1929, 1875, 1856 cm⁻¹; MS (EI+) *m/z* 170 (M⁺, 5), 157 (67), 131 (15), 84 (40), 57 (100); HRMS (EI+) *m/z* calcd for C₉H₁₈N₂O 170.1419, found 170.1420; Anal. Calcd for C₉H₁₈N₂O C, 63.49; H, 10.66; N, 16.45; Found C, 63.61; H, 10.79; N, 16.17.

21. (*E*)-1-Phenyl-1,3-butadiene was prepared according to a literature procedure: Grummitt, O.; Becker, E. I. *Org. Synth.* **1950**, *30*, 75; or Wittig, G.; Schoellkopf, U. *Org. Synth.* **1960**, *40*, 66. Similar results were obtained for step C using the diene prepared from either protocol.

22. Tetrakis(triphenylphosphine)palladium (9% min. palladium) was purchased from Pressure Chemical Co. and used as received.

23. Reaction is performed neat.

24. The submitters added di-*tert*-butyl-diaziridinone via a 10-mL addition funnel. The checkers observed more consistent results using a syringe pump.

25. Silica gel 40-63 D (60 Å) (Silicycle, Quebec City, Canada) was used.

26. Working at 50% scale, the checkers obtained 2.86 g (95%).

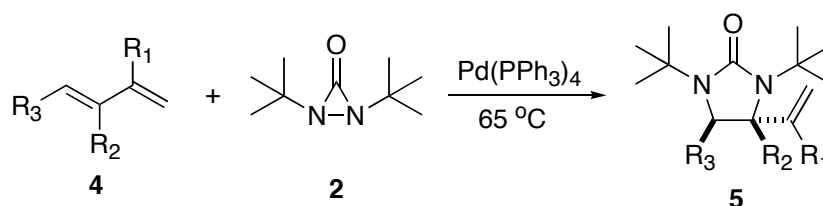
27. The product has the following physicochemical properties: ¹H NMR (300 MHz, benzene-*d*₆) δ: 1.36 (s, 9 H), 1.39 (s, 9 H), 3.55 (dm, *J* = 8.4 Hz, 1 H), 4.05 (d, *J* = 1.2 Hz, 1 H), 4.86 – 4.95 (m, 2 H), 5.92 (ddd, *J* = 17.1, 10.2, 8.4 Hz, 1 H), 7.07-7.16 (m, 3 H), 7.27 (dm, *J* = 6.9 Hz, 2 H); ¹³C NMR (75 MHz, benzene-*d*₆) δ: 29.1, 29.2, 53.7, 53.9, 63.8, 65.5, 115.6, 126.5, 129.4, 141.8, 145.1, 159.2; IR (ATR) 1682 cm⁻¹; MS (EI+) *m/z* 300 (M⁺, 55), 285 (100), 229 (81), 132 (60); HRMS (EI+) *m/z* calcd for C₁₉H₂₈N₂O 300.2202, found 300.2191; Anal. Calcd for C₁₉H₂₈N₂O C, 75.96; H, 9.39; N, 9.32; Found C, 76.01; H, 9.60; N, 9.31.

Safety and Waste Disposal Information

All hazardous materials should be handled and disposed of in accordance with “Prudent Practices in the Laboratory”; National Academy Press; Washington, DC, 1995.

3. Discussion

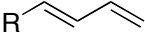
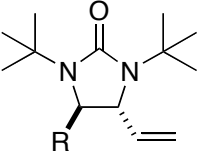
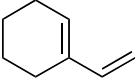
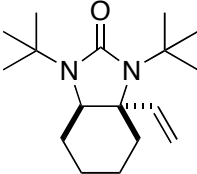
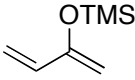
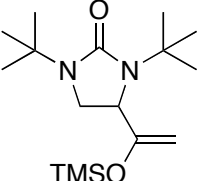
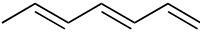
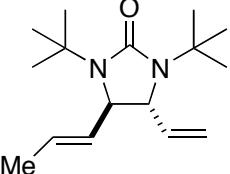
The diamination of olefins provides an attractive and efficient approach to biologically and chemically important vicinal diamines.⁵ To date, various metal-mediated⁶ or catalyzed⁷ diaminations have been developed. Recently, we reported that conjugated dienes and trienes can be regio- and stereoselectively diaminated using Pd(0)⁸ or Cu(I)⁹ as the catalyst and di-*tert*-butyldiaziridinone (**2**) or related compounds as a convenient nitrogen source. When Pd(0) is used as the catalyst, the diamination occurs regioselectively at the internal double bond (Scheme 1).^{8a}



Scheme 1

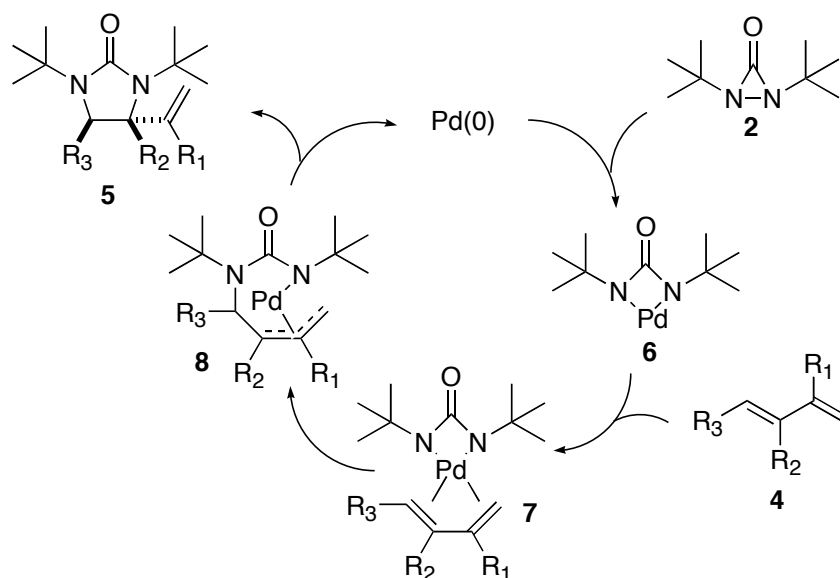
This diamination can be applied to various alkyl and aryl-substituted dienes as well as electron-rich and electron-deficient dienes (Table 1).^{8a} When the diamination is carried out under solvent-free conditions as described herein, the amount of Pd(PPh₃)₄ catalyst can be further reduced.¹⁰

Table 1. Pd(0)-Catalyzed Diamination of Conjugated Dienes and Trienes^a

Entry	Substrate (4)	Product (5)	Yield (%)
1	 R = Me		94
2	R = C ₅ H ₁₁		91
3	R = CH ₂ OBn		76
4	R = <i>p</i> -MeOPh		94
5	R = 2-Furyl		78
6	R = OMe		95
7	R = CO ₂ Me		62
8			86
9			90
10			86

^a All reactions were carried out with di-*tert*-butyldiaziridinone (**2**) (1.0 equiv), diene or triene (1.2 equiv), and Pd(PPh₃)₄ (0.1 equiv) in benzene-*d*₆ in an NMR tube at 65 °C under argon for 0.25 to 5 h.

A possible catalytic cycle for this diamination is shown in Scheme 2.^{8a} Oxidative insertion of Pd(0) into the N-N bond of diaziridinone **2** generates four-membered Pd(II) species **6**. This intermediate then reacts with diene **4** to form π -allyl Pd complex **8**, which undergoes a reductive elimination to release **5** and regenerate the Pd(0) catalyst.



Scheme 2

When a chiral ligand is used, the diamination proceeds with high enantioselectivity. Chiral diamines can be obtained upon deprotection and the double bonds contained in the diamination products can be elaborated into other functionalities.^{8d}

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2. Department of Chemistry, University of Pittsburgh, Pittsburgh, PA 15260, USA; pwipf@pitt.edu.
3. Greene, F. D.; Stowell, J. C.; Bergmark, W. R. *J. Org. Chem.* **1969**, *34*, 2254.
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5. For leading reviews, see: (a) Lucet, D.; LeGall, T.; Mioskowski, C. *Angew. Chem., Int. Ed.* **1998**, *37*, 2580. (b) Mortensen, M. S.; O'Doherty, G. A. *Chemtracts: Org. Chem.* **2005**, *18*, 555. (c) Kotti, S. R. S. S.; Timmons, C.; Li, G. *Chem. Biol. Drug Des.* **2006**, *67*, 101.
6. For metal-mediated diaminations, see: (a) Gomez Aranda, V.; Barluenga, J.; Aznar, F. *Synthesis* **1974**, 504. (b) Chong, A. O.; Oshima,

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10. The detailed studies will be described elsewhere.

Appendix

Chemical Abstracts Nomenclature; (Registry Number)

Di-*tert*-butylurea: Urea, *N,N'*-bis(1,1-dimethylethyl)-; (5336-24-3)
tert-Butylamine: 2-Propanamine, 2-methyl-; (75-64-9)
 Triethylamine: Ethanamine, *N,N*-diethyl-; (121-44-8)
 Triphosgene: Methanol, 1,1,1-trichloro-, 1,1'-carbonate; (32315-10-9)
 Di-*tert*-butyldiaziridinone: 3-Diaziridinone, 1,2-bis(1,1-dimethylethyl)-;
 (19656-74-7)

tert-Butyl-hypochlorite: Hypochlorous acid, 1,1-dimethylethyl ester; (507-40-4)
Potassium *tert*-butoxide: 2-Propanol, 2-methyl-, potassium salt (1:1); (865-47-4)
trans-1,3-Di-*tert*-butyl-4-phenyl-5-vinyl-imidazolidin-2-one: 2-Imidazolidinone, 1,3-bis(1,1-dimethylethyl)-4-ethenyl-5-phenyl-, (4*R*,5*R*)-rel-; (927902-91-8)
(*E*)-1-Phenyl-1,3-butadiene: Benzene, (1*E*)-1,3-butadienyl-; (16939-57-4)
Tetrakis(triphenylphosphine)palladium; (14221-01-3)



Yian Shi was born in Jiangsu, China in 1963. He obtained his B.Sc. degree from Nanjing University in 1983, M.Sc. degree from University of Toronto with Professor Ian W.J. Still in 1987, and Ph.D. degree from Stanford University with Professor Barry M. Trost in 1992. After a postdoctoral study at Harvard Medical School with Professor Christopher Walsh, he joined Colorado State University as assistant professor in 1995 and was promoted to associate professor in 2000 and professor in 2003. His current research interests include the development of new synthetic methods, asymmetric catalysis, and synthesis of natural products.



Haifeng Du was born in Jilin, China in 1974. He received his B.Sc. degree in 1998 and M.Sc. degree in 2001 from Nankai University. He then moved to Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, and obtained his Ph.D. degree in 2004 under the supervision of Professor Kuiling Ding. In the fall of 2004, he joined the Department of Chemistry at Colorado State University as a postdoctoral fellow with Professor Yian Shi. His research interests include the development of novel synthetic methodology and asymmetric synthesis.

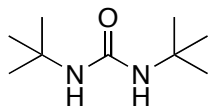


Baoguo Zhao was born in Hubei, China in 1973. He received his B.Sc. degree from Wuhan University in 1996 and M.Sc. degree from Nanjing University in 2002 under the supervision of Professor Jianhua Xu. After completing his Ph.D. degree under the supervision of Professor Kuiling Ding at Shanghai Institute of Organic Chemistry, Chinese Academy of Science in 2006, he joined Department of Chemistry at Colorado State University as a postdoctoral fellow with Professor Yian Shi. His current research interests include the development of novel synthetic methodology and asymmetric synthesis.



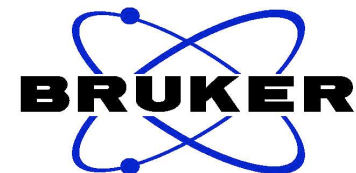
Eric Buck was born in 1985 in Fairmont, West Virginia. He graduated from the University of Minnesota with his B.Sc. in 2006. During his freshman year he entered the laboratory of Professor Thomas R. Hoye where he pursued the synthesis of petromyzonamine disulfate analogs with a focus on 5- β petromyzonamine disulfate. While attending the U of M he was supported by the David A. and Merece H. Johnson Scholarship. In 2007 he moved to Pittsburgh, Pennsylvania where he is currently a graduate student under the direction of Professor Peter Wipf. His current research interests include asymmetric synthesis and the synthesis of natural products.

di-tert-butylurea



— 157.02

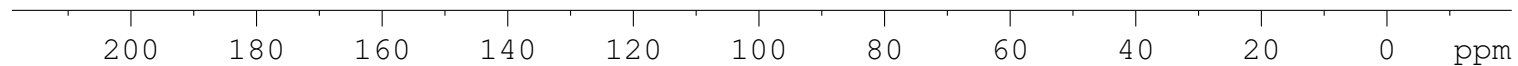
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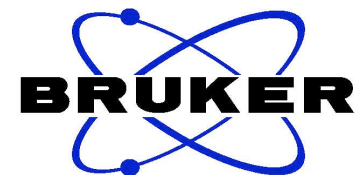
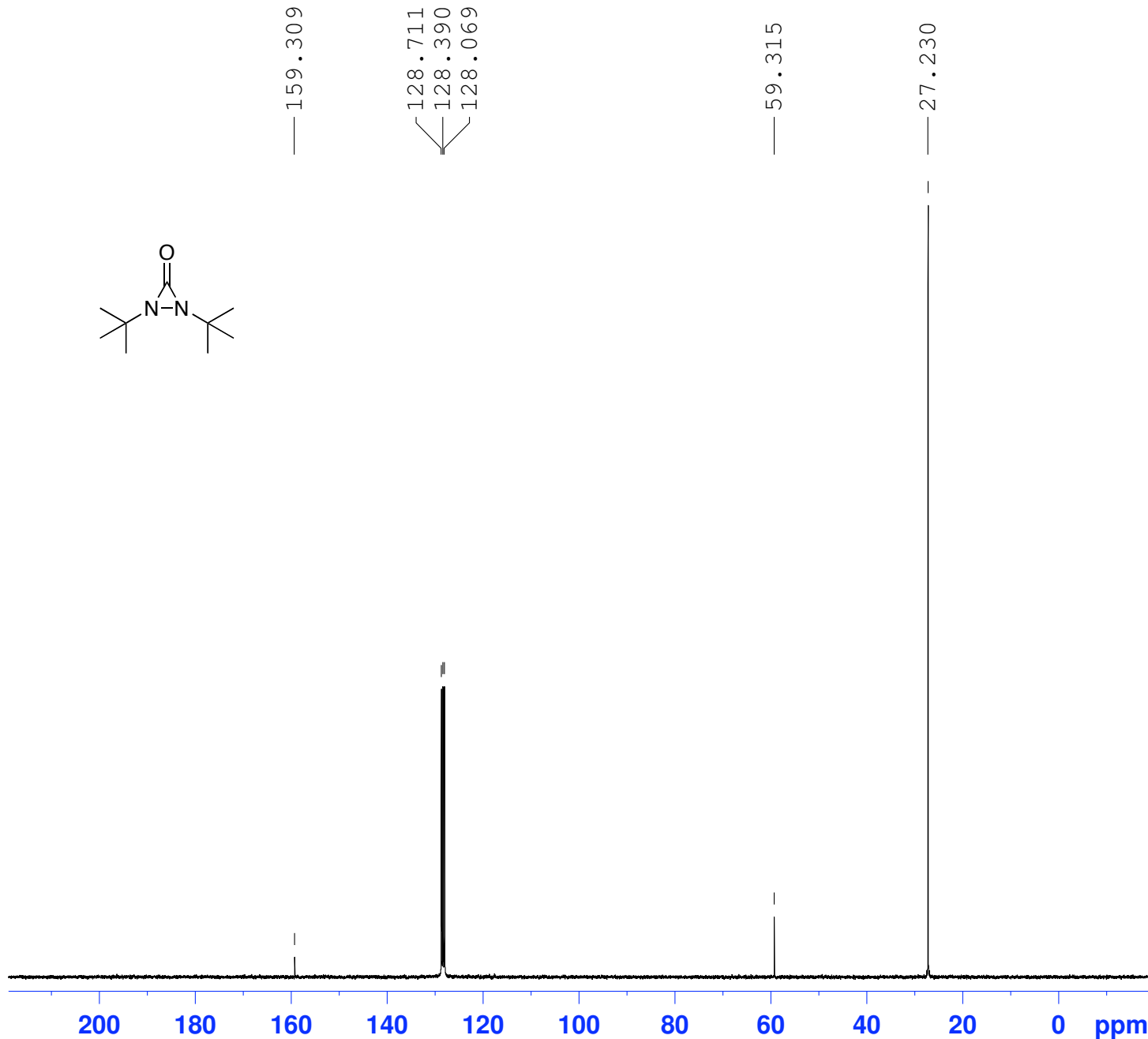
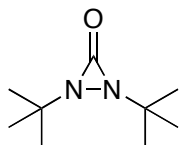
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di-tert-butyl diaziridinone

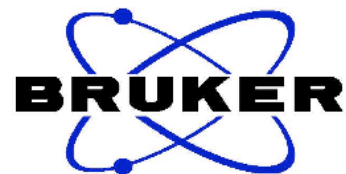


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RG 2050
DW 27.733 usec
DE 6.50 usec
TE 298.0 K
D1 5.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 2.00 dB
PL1W 16.99005508 W
SFO1 75.5004428 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -2.00 dB
PL12 15.51 dB
PL13 120.00 dB
PL2W 18.20942116 W
PL12W 0.32306963 W
PL13W 0.00000000 W
SFO2 300.2312009 MHz
SI 32768
SF 75.4928372 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

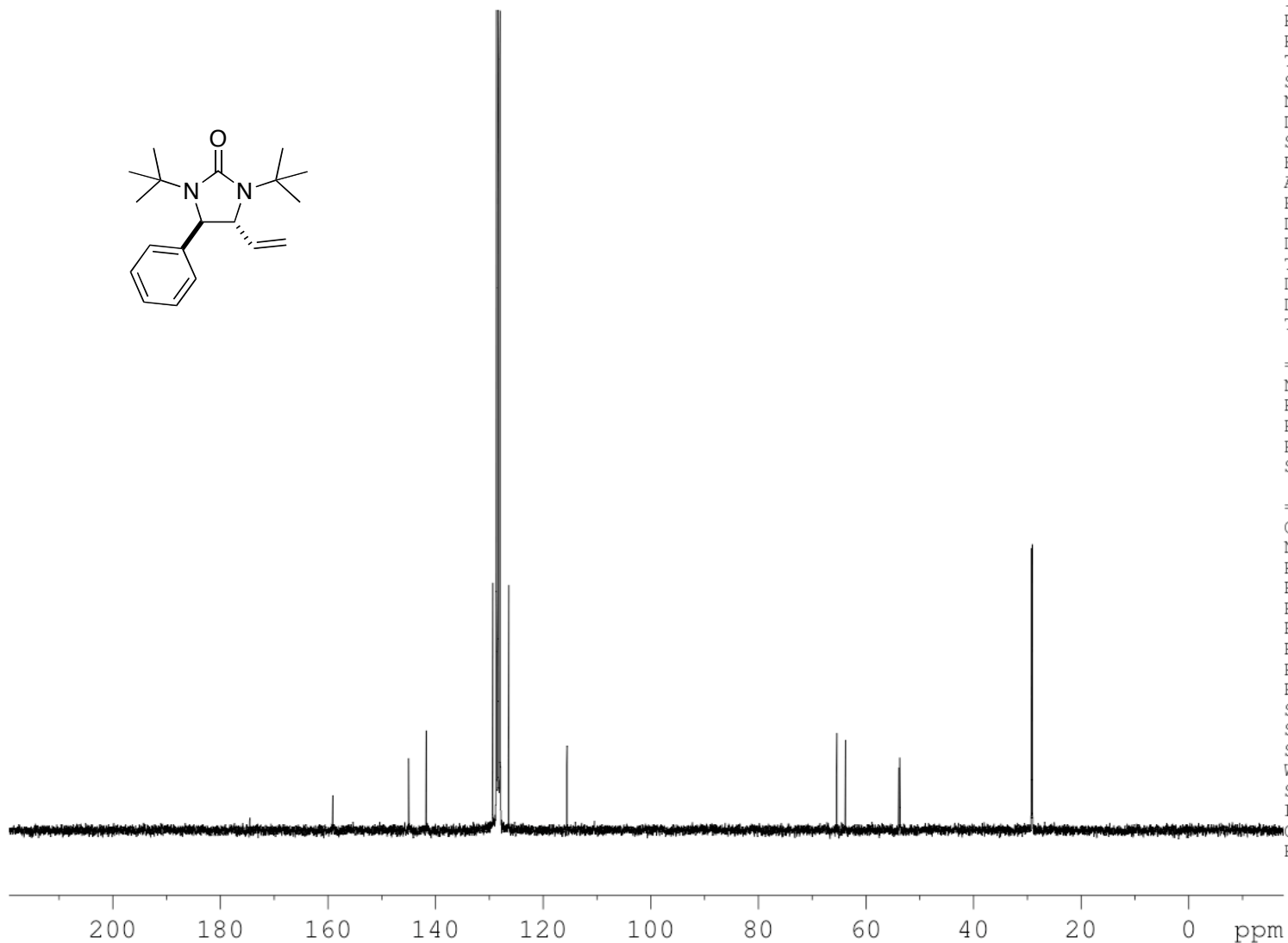
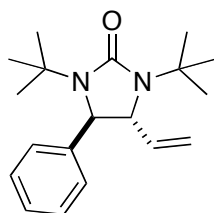
trans-1,3-Di-tert-butyl-4-phenyl-5-vinyl-imidazolidin-2-one 13C



— 159.15
 < 145.05
 < 141.77
 < 129.40
 < 128.71
 < 128.39
 < 128.07
 < 126.45
 < 115.61

 < 65.45
 < 63.82
 < 53.91
 < 53.70

 < 29.23
 < 29.09

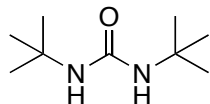


NAME 156.094-13C
 EXPNO 2
 PROCNO 1
 Date_ 20090225
 Time 11.06
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zgpg
 TD 65536
 SOLVENT C6D6
 NS 54
 DS 4
 SWH 18028.846 Hz
 FIDRES 0.275098 Hz
 AQ 1.8175818 sec
 RG 2050
 DW 27.733 usec
 DE 6.50 usec
 TE 293.8 K
 D1 5.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.00 usec
 PL1 2.00 dB
 PL1W 16.99005508 W
 SFO1 75.5004428 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 -2.00 dB
 PL12 15.51 dB
 PL13 120.00 dB
 PL2W 18.20942116 W
 PL12W 0.32306963 W
 PL13W 0.00000000 W
 SFO2 300.2312009 MHz
 SI 32768
 SF 75.4928397 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

di-tert-butylurea



— 5.445

— 2.500

— 1.184



```
NAME      di-tert-butylurea-1H
EXPNO     3
PROCNO    1
Date_     20090225
Time      13.27
INSTRUM   spect
PROBHD    5 mm Multinucl
PULPROG   zg
TD         32768
SOLVENT   DMSO
NS         8
DS         2
SWH        6188.119 Hz
FIDRES     0.188846 Hz
AQ         2.6477044 sec
RG         4
DW         80.800 usec
DE         6.50 usec
TE         292.9 K
D1         1.00000000 sec
TD0        1
```

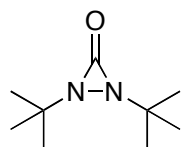
```
===== CHANNEL f1 =====
NUC1      1H
P1         9.00 usec
PL1        -2.00 dB
PL1W      18.20942116 W
SFO1      300.2318540 MHz
SI         32768
SF         300.2299998 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
```



2.04

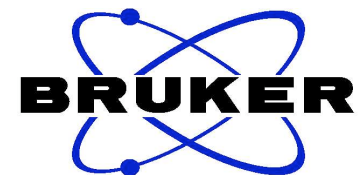
18.00

di-tert-butyl diaziridinone



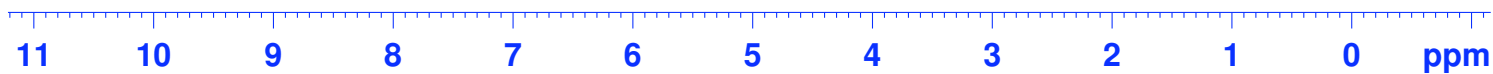
— 7.160

— 1.068



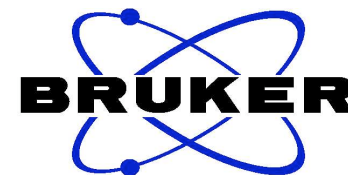
NAME 156.069-2
EXPNO 1
PROCNO 1
Date_ 20090219
Time 16.02
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT C6D6
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 4
DW 80.800 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 -2.00 dB
PL1W 18.20942116 W
SFO1 300.2318540 MHz
SI 32768
SF 300.2299925 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

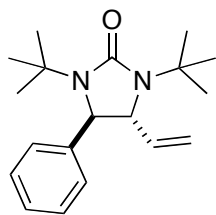


18.00

trans-1,3-di-tert-butyl-4-phenyl-5-vinyl-imidazolidin-2-one



7.283
7.277
7.271
7.254
7.251
7.160
7.140
7.125
7.120
7.096
7.091
7.086
7.067
5.979
5.951
5.945
5.922
5.917
5.894
5.888
5.860
4.950
4.946
4.943
4.898
4.896
4.893
4.890
4.888
4.886
4.866
4.865
4.862
4.053
4.049
3.564
3.562
3.560
3.536
3.534
3.532
1.394
1.355



```

NAME          156.094_3
EXPNO         1
PROCNO       1
Date_        20090227
Time         20.05
INSTRUM     spect
PROBHD      5 mm Multinucl
PULPROG     zg
TD          32768
SOLVENT     C6D6
NS          16
DS          2
SWH         6188.119 Hz
FIDRES     0.188846 Hz
AQ         2.6477044 sec
RG          4
DW         80.800 usec
DE         6.50 usec
TE         292.9 K
D1         1.00000000 sec
TD0        1
    
```

```

===== CHANNEL f1 =====
NUC1         1H
P1           9.00 usec
PL1         -2.00 dB
PL1W        18.20942116 W
SFO1        300.2318540 MHz
SI          32768
SF          300.2299923 MHz
WDW         EM
SSB         0
LB          0.30 Hz
GB          0
PC          1.00
    
```

