Thermolysis of Novel Free Radical Initiators: t-Butylazocumene and its 1,3- and 1,4-Bisazo and 1,3,5-Trisazo Analogs

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Supplementary Material

Kinetic Treatment of 7.

CKS was first used to simulate the "first order" plots of nitrogen evolution for the system

\[
\begin{align*}
C & \xrightarrow{k_1} D + N_2 \\
D & \xrightarrow{k_2} E + N_2 \\
E & \xrightarrow{k_3} N_2
\end{align*}
\]

where C represents 7, D is the meta bisazoalkane from 7, and E is the monoazoalkane from D. The resulting curves were similar to those shown in Fig. 1 for 5 but this time the plot was perfectly linear when \(k_1: k_2: k_3\) were in the ratio 3:2:1. This system of three differential equations was again solved analytically, and values of \(k_1, k_2,\) and \(k_3\) were extracted by fitting the experimental pressure versus time data to eq. (2) using Kaleidagraph. The resulting values of \(k_1\) were fitted to the Eyring equation to obtain the activation parameters in Table 2.

\[
\ln (P_\infty - P) = \ln \left\{ 3[C]_0 \exp(-k_1t) + 2[[C]_0 k_1(k_2-k_1)^{-1}[\exp(-k_1t) - \exp(-k_2t)]\right\} + \\
\left[[C]_0 k_1k_2(k_2-k_1)^{-1}[(k_3-k_1)^{-1}\exp(-k_1t) - (k_3-k_2)^{-1}\exp(-k_2t)] + \\
[C]_0 k_1k_2(k_3-k_2)^{-1}(k_3-k_1)^{-1}\exp(-k_3t)\right\}
\]

where \([C]_0 = (P_\infty - P_0)/3\).
Synthesis of Compounds

Cumylamine 8 was prepared according to Ciganek\(^1\) and Imamoto\(^2\) in 90% yield. \(^1\)H NMR δ 1.504 (s, 6H), 1.71 (s, 2H), 7.190-7.532 (m, 5H); \(^13\)C NMR δ 32.75, 52.31, 124.58, 126.09, 128.12, 150.39; HRMS (70 ev, EI) calcd for C\(_9\)H\(_{13}\)N 135.1048, found 135.1048.

Cumyl-t-butylsulfamide 9. Two equivalents of cumylamine in 10 mL of anhydrous ether was added dropwise to a solution of t-butylsulfamyl chloride\(^3\) (26 mmol) in 50 mL hexane at 0 °C under nitrogen. The mixture was stirred for 1.5 h at room temperature and was quenched by addition of water. After filtration, the solid was washed with 5% aqueous sodium bicarbonate. The yield was 95% based on consumed cumylamine. Unreacted cumylamine was recovered from the aqueous phase by basifying the solution with conc. NaOH and extracting with CH\(_2\)Cl\(_2\). \(^1\)H NMR δ 1.26 (s, 9H), 1.75 (s, 6H), 4.77 (br s, 2H), 7.23-7.53 (m, 5H); \(^13\)C NMR δ 29.26, 29.78, 54.20, 58.21, 125.54, 127.16, 128.33, 146.08.

t-Butylazocumene 4. Cumyl-t-butylsulfamide (1.0 g) was suspended in 15 mL of 50% of ice cold aq. NaOH (1.2 g, 8 equivalents). Commercially available bleach (44 mL 5% NaOCl, 8 equiv.) was added to the cold base solution over one h. The mixture was allowed to warm to room temperature and was stirred overnight. The aqueous solution was extracted with 3 x 50 mL hexane, which was dried over Na\(_2\)SO\(_4\) and rotary evaporated. The product was purified by silica gel chromatography with 5% ethyl acetate in hexane as eluent. Yield 90%. \(^1\)H NMR δ 1.22 (s, 9H), 1.47 (s, 6H), 7.25-7.41 (m, 5H); \(^13\)C NMR δ 26.56, 26.78, 66.58, 70.75, 126.10, 126.40, 128.11, 146.54; UV (pentane) λ\(_{max}\) = 366 nm, ε = 26.

1,4-Bis-(2-amino-2-propyl)benzene was obtained by the Ciganek method\(^1\) using 0.167 equivalents of 1,4-dicyanobenzene. The mixture was stirred for 3 h in a -65 °C bath, and then for 4 h at -25 °C. The pure bisamine was obtained as a white solid in 95% yield. \(^1\)H NMR δ 1.49 (s, 12H), 1.65 (br s, 4H), 7.46 (s, 4H); \(^13\)C NMR δ 32.70, 52.04, 124.43, 148.1; HRMS (CI) calc. for C\(_{12}\)H\(_{21}\)N\(_2\) (M+H) 193.1705; found 193.1701.

1,4-Bis-(2-t-butylsulfamido-2-propyl)benzene was made from the above diamine by the same method as for cumyl-t-butylsulfamide, except that the molar ratio of diamine to t-
butylsulfamyl chloride was 1:1. Unreacted diamine was recovered from the aqueous phase by basifying the solution with conc. NaOH and extracting with CH\textsubscript{2}Cl\textsubscript{2}. Yield: 90\% based on the consumed diamine. \textsuperscript{1}H NMR \(\delta\) 1.30 (s, 18H), 1.75 (s, 12H), 3.80 (s, 2H), 4.44 (s, 2H), 7.49 (s, 4H); \textsuperscript{13}C NMR (DMSO) \(\delta\) 29.29, 29.68, 52.66, 57.03, 124.84, 146.18.

\textbf{1,4-Bis-(2-t-butylazo-2-propyl)benzene} \(\text{5}\) was made and purified similarly to \(\text{4}\), except the oxidation time was 24 h. After recrystallization from MeOH, the product was obtained as light yellow flakes in 86\% yield. mp 77-79 °C. \textsuperscript{1}H NMR \(\delta\) 1.21 (s, 18H), 1.44 (s, 12H), 7.33 (s, 4H); \textsuperscript{13}C NMR \(\delta\) 26.58, 26.80, 66.56, 70.57, 125.85, 144.55; UV (pentane) \(\lambda_{\text{max}} = 366\) nm, \(\varepsilon = 59\); HRMS (CI) calcld for C\textsubscript{20}H\textsubscript{35}N\textsubscript{4} (M+H) 331.2862; found 331.2856.

\textbf{1,3-Bis-(2-amino-2-propyl)benzene} was made in 80\% yield by the same method as for the para isomer. \textsuperscript{1}H NMR \(\delta\) 1.50 (s, 12H), 1.73 (s, 4H); 7.25-7.36 (m, 3H), 7.67-7.69 (m, 1H); \textsuperscript{13}C NMR \(\delta\) 32.88, 52.54, 120.84, 122.56, 127.91, 150.09.

\textbf{1,3-Bis-(2-t-butylsulfamido-2-propyl)benzene} was made from the 1,3-diamine just as in the case of the para isomer. This sulfamide decomposed on silica gel and attempts to recrystallize it at low temperature from MeOH or hexane also failed. The bis-sulfamide was finally purified by washing several times with ether. Yield 41.5\% \textsuperscript{1}H NMR \(\delta\) 1.22 (s, 18H), 1.74 (s, 12H), 4.70 (br s, 2H), 4.85 (br s, 2H), 7.33 (br s, 3H), 7.92 (br s, 1H); \textsuperscript{13}C NMR \(\delta\) 29.70, 29.83, 54.44, 57.99, 123.94, 124.20, 128.03, 145.64.

\textbf{1,3-Bis-(2-t-butylazo-2-propyl)benzene} \(\text{6}\) was synthesized by the same method as \(\text{5}\). The product was purified by recrystallization from MeOH at -5 °C. Yield 72\%. mp 31-32 °C. \textsuperscript{1}H NMR \(\delta\) 1.22 (s, 18H), 1.44 (s, 12H), 7.24-7.36 (m, 4H); \textsuperscript{13}C NMR \(\delta\) 26.58, 26.80, 66.51, 70.94, 124.10, 124.32, 127.85, 146.11; UV (hexane) \(\lambda_{\text{max}} = 366\) nm, \(\varepsilon = 66.5\). HRMS (FAB) calcld for C\textsubscript{20}H\textsubscript{35}N\textsubscript{4} (M+H) 331.2862, found 331.2870.

\textbf{1,3,5-Tricyanobenzene} \(\text{10}\) was prepared by dehydration of benzene-1,3,5-tricarboxamidine.\textsuperscript{4} Yield 78\%. mp 256-258 °C \textsuperscript{1}H NMR \(\delta\) 8.18 (s, 3H); \textsuperscript{13}C NMR \(\delta\) 114.60, 115.92, 138.66. IR 3090.5, 2253, 1736, 1644, 1422 cm\textsuperscript{-1}. 

3
1,3,5-Tris-(2-amino-2-propyl)benzene 12 was made by the same procedure as 8 except that 0.111 equivalent of 1,3,5-tricyanobenzene was used relative to the cerium reagent, and the reaction was run at -65 °C for 2 h and -25 to -30 °C for 4 h. The product consisted of 60% trisamine 12 and 40% monocyanoobisamine 11. To avoid separating these amines, the crude product was treated again with 3-4 equivalents of cerium reagent. The same workup procedure yielded a thick, yellow liquid in 65% yield. The pure 12 was obtained by recrystallization from 30% ether in pentane at -21 °C. $^1$H NMR δ 1.52 (s, 18H), 7.53 (s, 3H); $^{13}$C NMR δ 33.01, 52.72, 118.99, 149.86; HRMS (CI) calcd for C$_{15}$H$_{27}$N$_3$ 250.2283 (M+H); found 250.2279.

1,3,5-Tris-(2-t-butylsulfamido-2-propyl)benzene was prepared in the same manner as the cumyl analog 9. The molar ratio of trisamine 12 to t-butylsulfamyl chloride was 1:1.5. The trisulfamide was purified by washing with ether several times. Yield: 80% based on consumed 12. mp 190-191 °C. $^1$H NMR δ 1.25 (s, 27H), 1.76 (s, 18H), 7.63 (s, 3H).

1,3,5-Tris-(2-t-butylazo-2-propyl)benzene 7 was made in basic bleach solution as in the case of 4, except that 10% v/v CH$_2$Cl$_2$ was added and the reaction time was 3 days. Yield: 60% of 7 as a thick oil. Attempted purification by short path distillation, column chromatography, or preparative TLC led to decomposition while recrystallization failed to give a solid. $^1$H NMR δ 1.22 (s, 27H), 1.42 (s, 18H), 7.20 (s, 3H); $^{13}$C NMR δ 26.65, 26.83, 66.45, 71.18, 122.19, 145.62; UV (pentane) $\lambda_{max}$ = 362 nm, $\varepsilon = 69$. HRMS (CI) calcd for C$_{27}$H$_{48}$N$_6$ (M+H) 457.4019; found 457.3994.

1-Isopropenyl-4-cyanobenzene. Methylthiium (1.2 equiv., 4 mL, 1.4 M) was slowly added to a suspension of methyltriphosphonium bromide (1.2 equiv., 2.0 g) in 12 mL ether at 0 °C. The yellow mixture was stirred for 20 min at room temperature, 4-acetylbenzonitrile (1 equiv., 0.70 g in 5 mL THF) was added at 0 °C, and the reaction mixture was stirred for 2.5 h at room temperature. Water (3 mL) was added to quench the reaction. The product was purified by silica gel chromatography using 25% ethyl acetate in hexane as eluent. Yield 53%. $^1$H NMR δ 2.16 (s, 3H), 5.24-5.25 (m, 1H), 5.47 (s, 1H), 7.53 (d, $J = 9.7$ Hz, 2H).
2H), 7.61 (d, J = 9.7 Hz, 2H); $^{13}$C NMR δ 21.38, 110.78, 115.59, 118.91, 126.06, 132.03, 141.73, 145.58. HRMS (EI) calcd for C$_{10}$H$_9$N 143.0735, found 143.0733.

1-Isopropenyl-4-(2-amino-2-propyl)benzene was obtained by the Ciganek method as described in the synthesis of 8. The reaction time was 4 h. The product was purified by silica gel chromatography, eluting with 30% ethyl acetate in hexane. Yield 88%. $^{1}$H NMR δ 1.51 (s, 6H), 2.15 (s, 3H), 5.06-5.07 (m, 1H), 5.36-5.37 (m, 1H), 7.45 (d, J = 2.9 Hz, 3H), 7.46 (d, J = 2.9 Hz, 2H); $^{13}$C NMR δ 21.75, 32.45, 52.42, 112.06, 124.55, 125.31, 139.16, 142.80, 149.06; HRMS (EI) calcd for C$_{12}$H$_{17}$N 175.1359; found 175.1361.

1-Isopropenyl-4-(2-t-butylsulfamido-2-propyl)benzene was made by the same method as cumyl-t-butylsulfamide 9. The product was purified by silica gel chromatography using 35% ethyl acetate in hexane as eluent. $^{1}$H NMR δ 1.2 (s, 9H), 1.7 (s, 6H), 2.2 (s, 3H), 3.85 (s, 1H), 4.8 (s, 1H), 5.2 (s, 1H), 5.5 (s, 1H), 7.5 (br, s, 4H); HRMS (CI) calcd for C$_{16}$H$_{26}$N$_2$SO$_2$ 311.1793 (M+H); found 311.1791.

1-Isopropyl-4-(2-t-butylsulfamido-2-propyl)benzene (1-Isopropenyl-4-(2-t-butyl-sulfamido-2-propyl)benzene (0.207 g) was dissolved in 50 mL MeOH and about 5 mg of 10% Pd/C was added. The mixture was hydrogenated at 1 atm. for 1 h at room temperature. The solution was filtered and solvent was removed by rotary evaporation. $^{1}$H NMR δ 1.24 (d, J = 6.9 Hz, 6H), 1.25 (s, 9H), 1.75 (s, 6H), 2.90 (septet, J = 6.9 Hz, 1H), 3.61 (s, 1H), 4.42 (s, 1H), 7.22 (d, J = 8.2 Hz, 2H), 7.43 (d, J = 8.2 Hz, 2H); $^{13}$C NMR δ 23.92, 29.24, 29.76, 33.60, 54.15, 58.00, 125.60, 126.41, 143.15, 147.9. HRMS (CI) calcd for C$_{16}$H$_{28}$N$_2$SO$_2$ 313.1950 (M+H); found 313.1951.

1-Isopropyl-4-(2-t-butyrazo-2-propyl)benzene was made and purified similarly to t-butyrazocumene 4. Yield 76%.$^{1}$H NMR δ 1.21 (s, 9H), 1.25 (d, J = 6.8 Hz, 6H), 1.43 (s, 6H), 2.90 (septet, J = 6.8 Hz, 1H), 7.20 (d, J = 8.3 Hz, 2H), 7.29 (d, J = 8.3 Hz, 2H); $^{13}$C NMR δ 23.98, 26.59, 26.80, 33.64, 66.49, 70.57, 125.97, 126.15, 143.84, 146.78. HRMS (CI) calcd for C$_{16}$H$_{26}$N$_2$ 247.2174 (M+H), found 247.2176.
1,3,5-Tris-[2-(2,2,6,6-tetramethylpiperidin-1-yl)-2-propyl]benzene 32b. A solution of trisazoalkane 7 (10 mg) and TEMPO (6 equiv., 20.5 mg) in 0.5 mL benzene was degassed, sealed, and irradiated at 366 nm for 2 h. The product was isolated by preparative TLC using hexane as eluent. $^1$H NMR $\delta$ 0.829-1.42 (m, 18H), 0.829 (s, 18H), 1.10 (s, 18H), 1.58 (s, 18H), 7.42 (s, 3H). Because 32b partially decomposed on standing at room temperature overnight, this trialkylhydroxylamine was not characterized further.

1,4-Bis-[2-(2,2,6,6-tetramethylpiperidin-1-yl)-2-propyl]benzene 35. Bisazoalkane 5 (10 mg) and TEMPO (4 equiv., 18.9 mg) were dissolved in 0.5 mL benzene. The solution was freeze-thaw degassed three times, sealed under vacuum, and irradiated at 366 nm for 2 h. The product was isolated by preparative TLC using hexane as eluent. $^1$H NMR $\delta$ 0.792 (s, 12H), 0.853-1.44 (m, 12H), 1.05 (s, 12H), 1.59 (s, 12H), 7.37 (s, 4H). Some of 35 decomposed in CDCl$_3$ on standing at room temperature overnight, but the pure compound seemed stable at -5 °C. 35 was also made from p-diisopropylbenzene with di-t-butylhydroxymethyl instead of di-t-butyl peroxide as initiator.$^7$ 1,4-Diisopropylbenzene (80 mg), 5 equiv. of TEMPO and 5 equiv. of di-t-butylhydroxymethyl were dissolved in 2.5 mL of acetonitrile in a quartz tube. After degassing, the solution was subjected to 254 nm irradiation for 23 h. The product 35 was isolated by silica gel chromatography using hexane as eluent.

di-Heptylbenzene (1,1,2,2-tetramethyl-1-phenylpropane) was isolated from the thermolysis products of 4 by preparative GC. The separation conditions were: Inj. 160 °C, det. 200 °C, column 174 °C, flow rate 50 mL/min., column 20% OV101 on Ch. W. 1/4" x 10'. $^1$H NMR $\delta$ 0.844 (s, 9H), 1.352 (s, 6H), 7.162-7.387 (m, 5H); $^{13}$C NMR $\delta$ 24.37, 26.33, 35.87, 42.86, 125.19, 126.86, 128.32, 147.68.

Bicumyl was isolated from the thermolysis of azocumene by preparative TLC using hexane as eluent. $^1$H NMR $\delta$ 1.314 (s, 12H), 7.05-7.20 (m, 10H).

1-tert-Butyl-4-isopropylbenzene 14 was synthesized in 66% yield by alkylation of cumene.$^8$ $^1$H NMR $\delta$ 1.25 (d, J=6.9 Hz, 6H), 1.31 (s, 9H), 2.89 (septet, J=6.9 Hz, 1H), 7.17 (d, J = 8.17 Hz, 2H), 7.32 (d, J = 8.17 Hz, 2H).
2-Phenyl-2-(thiophenyl)propane 20. Isopropenylbenzene (0.20 g) and thiophenol (2 equiv., 0.273 g) were dissolved in 10 mL CH₂Cl₂. Trifluoroacetic acid (1 mL) was added dropwise to solution, which was cooled to -10 °C. The cold bath was removed and the solution was stirred for 2 h at 25 °C. At 0 °C, 5 mL of saturated NH₄Cl solution was added slowly to the reaction mixture. The organic layer was separated and washed twice with 25% NaOH solution and three times with brine. The sulfide was purified by silica gel column chromatography using hexane as eluent. Yield 78%. ¹H NMR δ 1.693 (s, 6H), 7.14-7.43 (m, 10H).

2-(4-Isopropylphenyl)-2-(thiophenyl)propane 24 was prepared by the method immediately above starting from 1-isopropyl-4-isopropenylbenzene. ¹H NMR δ 1.246 (d, J = 6.9 Hz, 6H), 1.667 (s, 6H), 2.884 (septet, J = 6.9 Hz, 1H), 7.113-7.350 (m 9H). ¹³C NMR δ 23.97, 29.66, 33.58, 50.83, 125.87, 126.39, 128.15, 128.42, 132.99, 136.59, 143.53, 147.13. HRMS (Cl) calc. for C₁₈H₂₂S (M+H) 271.1520; found 271.1517.

2-Phenyl-1-(thiophenyl)propane 21 was made by the method of Screttas.¹¹ ¹H NMR δ 1.39 (d, J=6.8 Hz, 3H), 2.93-3.08 (m, 2H), 3.20-3.24 (dd J = 6.2, 12.5 Hz, 1H), 7.19-7.32 (m, 10H). ¹³C NMR δ 20.98, 39.42, 42.03, 125.80, 126.56, 126.93, 128.50, 128.84, 129.09, 136.81, 145.49.

2-(4-Isopropylphenyl)-1-(thiophenyl)propane 25 was prepared as immediately above starting with 1-isopropyl-4-isopropenylbenzene. The product was purified by silica gel column chromatography using hexane as eluent. Yield 80%. ¹H NMR δ 1.24 (d, J=6.8 Hz, 6H), 1.38 (d, J=6.7 Hz, 3H), 2.86-3.05 (m, 3H), 3.19-3.24 (m, 1H), 7.13-7.29 (m, 9H). ¹³C NMR δ 20.89, 23.98, 33.65, 39.00, 42.09, 125.70, 126.49, 126.78, 128.79, 128.98, 136.93, 142.81, 146.99.

9-Azabicyclo[3.3.1]nonane-N-oxyl (ABNO) was made according to the literature.¹² The product was purified by sublimation under vacuum in a 83 °C oil bath. λmax = 483, ε = 8.8 in hexane; lit. ε = 8.5 in cyclohexane.¹³

2-(9-Azabicyclo[3.3.1]nonane-N-oxyl)-2-phenylpropane 29. A solution of 4 and two equiv. of ABNO in 0.5 mL benzene was degassed, sealed, and heated at 124 °C for 35
min. The solvent was removed under vacuum and the product was isolated by preparative TLC using hexane as eluent. $^1$H NMR $\delta$ 1.20-2.10 (m, 10H), 1.54 (s, 6H), 2.31 (m, 2H), 2.95 (br s, 2H), 7.16-7.34 (m, 3H), 7.49-7.54 (m, 2H); $^{13}$C $\delta$ 19.933, 20.113, 23.274, 26.781, 31.871, 55.604, 79.515, 125.659, 126.384, 127.614, 147.632.

1,3,5-Tris-(2-(9-azabicyclo[3.3.1]nonane-N-oxyl)-2-propyl)benzene 32a. A solution of 7 (35 mg, 0.077 mol) and ABNO (37 mg, 0.26 mmol) in 0.5 mL of benzene was degassed, sealed, and heated at 124 °C for 35 min. The solvent was removed under vacuum and the product was separated by preparative TLC using hexane as eluent. Yield 18%. $^1$H NMR $\delta$ 1.19-1.99 (m, 36H), 1.54 (s, 18H), 2.33-2.41 (m, 6H), 2.94 (br s, 6H), 7.53 (s, 3H); $^{13}$C $\delta$ 20.00, 20.18, 23.24, 26.94, 31.89, 55.51, 79.79, 121.48, 145.68. HRMS (CI) for C$_{39}$H$_{64}$N$_3$O$_3$ (M+H) calc. 622.4948; found 622.4946.

### Additional Tables

**Table 8. Kinetic Modelling of Scheme 1a**

<table>
<thead>
<tr>
<th>$10^4 k_2$</th>
<th>$B_{max}$</th>
<th>$10^4 m$</th>
<th>$R^e$</th>
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<td>10.0</td>
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- **a.** See figure 1. $[A]_0 = 0.05$ M, $k_1 = 2.0 \times 10^{-4}$ s$^{-1}$.  
  - **b.** Assumed rate constant (s$^{-1}$) for second step of Scheme 1.
  - **c.** Maximum concentration (M) reached by monoazo intermediate 16 in Scheme 1.  
  - **d.** $m = \text{slope} = \text{apparent rate constant (s}^{-1}\text{)}$ for N$_2$ evolution using all data points.  
  - **e.** Correlation coefficient.
Table 9. Thermolysis Products of 7 with PhSH\textsuperscript{a}

<table>
<thead>
<tr>
<th>Product\textsuperscript{b}</th>
<th>ret'n time, min\textsuperscript{c}</th>
<th>moles/mole 7\textsuperscript{d}</th>
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<tbody>
<tr>
<td>isobutane\textsuperscript{e}</td>
<td>1.69</td>
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<tr>
<td>PhSH\textsuperscript{e}</td>
<td>9.56</td>
<td>5.93</td>
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<td>n-dodecane\textsuperscript{g}</td>
<td>14.08</td>
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<td>phenyl-t-butyl sulfide\textsuperscript{e}</td>
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\textsuperscript{a} 6.6 mg 7 + 1.13 mg dodecane + 20.7 mg PhSH in 0.25 mL C\textsubscript{6}D\textsubscript{6}, heated at 112.9 °C for 8 h. \textsuperscript{b-e, g, i} See footnotes to Table 4.
Table 10. Thermolysis products of 5 without radical scavenger\textsuperscript{a}

<table>
<thead>
<tr>
<th>Product\textsuperscript{b,k}</th>
<th>ret’n time,\textsuperscript{c} min</th>
<th>moles/mole \textsuperscript{d}</th>
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<td>2,2,3,3-tetramethylbutane\textsuperscript{f}</td>
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<td>\includegraphics[width=0.1\textwidth]{diagram.png} \textsuperscript{17l}</td>
<td>23.14</td>
<td>0.0971</td>
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<td>\includegraphics[width=0.1\textwidth]{diagram.png} \textsuperscript{i, m}</td>
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<td>\includegraphics[width=0.1\textwidth]{diagram.png} \textsuperscript{i} \textsuperscript{2}</td>
<td>33.83</td>
<td>0.0134</td>
</tr>
</tbody>
</table>

\textsuperscript{a} 4.3 mg 5 + 1.13 mg dodecane in 0.25 mL C\textsubscript{6}D\textsubscript{6} heated at 112.9\degree C for 8 h. \textsuperscript{b-g, i} see footnotes to Table 4. \textsuperscript{k} unidentified peaks below 0.01 moles/mole at 21.37, 22.13, 26.52 min. \textsuperscript{l} Same retention time as the major thermolysis product of 1-isopropyl-4-(2-t-butylazo-2-propyl)benzene. \textsuperscript{m} Could also be 4-t-heptyl-2,5-di-t-butylcumene.
References


