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Supporting Information

For

Dynamic Solvent Effect in Radical–Radical Coupling Reactions: An Almost Bottleable Localized Singlet Diradical

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1. General Experimental Information

AZ1a-d were synthesized according to the literature procedure.^[1] Solvents used for laser flash photolysis (LFP) measurements were purchased from commercial suppliers and used without further purification. The concentration of samples for LFP measurements under atmospheric pressure was adjusted to 5 mM and its optical density were around 0.4 at the excited wavelength (355 nm). The excitation source for the LFP system was an Nd:YAG laser ($\lambda_{exc} = 355$ nm, 5 ns pulse, 7 mJ). The monitoring system consisted of a 150 W xenon lamp as light source, Unisoku-MD200 monochromator and a photomultiplier. The temperature was controlled with a CoolSpek USP-203-B (Unisoku). The concentration of samples for LFP measurements under high pressure was adjusted to 10 mM. Details of high pressure kinetics were described elsewhere ^[2] while Nd:YAG laser, 150 W xenon lamp and Unisoku-MD200 were used as excitation source, light source and monochromator, respectively. The solvent polarity parameter, π^* and $E_T(30)$, were cited from corresponding articles.^[3,4] And π^* for Methyl cyclohexane and 2,4-dicyclohexyl-2-methyl-pentane were determined by the measurements of UV-vis absorption of 4-nitro-anisole (vide infra).The solvent viscosity, η , were cited from corresponding articles. ^[5-13] Pressure dependency of solvent viscosity for glycerol triacetate and 2,4-dicyclohexyl-2-methyl-pentane were calculated from corresponding articles using $\eta = \eta_0$ exp (α P).^[12,13] And that for methyl cyclohexane were estimated from previous report (vide infra).

2. Transient Absorption spectra

Transient Absorption spectra of **AZ1a-d** were measured at around 293 K in each solvent under N₂ atmosphere. In all cases, transient species, which has absorption maximum at around 560 – 600 nm, were observed like Figure S1-S4. And these absorptions were corresponding to HOMO-LUMO transition of S-**DR1a-d**.



Figure S1. Transient Absorption of S-DR1a in hexane at 294.1 K (blue: 0.02 microseconds, yellow: 0.15 microseconds, orange: 0.3 microseconds, gray: 0.6 microseconds)



Figure S2. Transient Absorption of S-DR1b in hexane at 295.5 K (blue: 0.03 microseconds, yellow: 0.5 microseconds, orange: 0.1 microseconds, gray: 0.6 microseconds)



Figure S3. Transient Absorption of S-DR1c in hexane at 291.7 K (blue: 0.1 microseconds, yellow: 2.0 microseconds, orange: 4 microseconds, gray: 8 microseconds)



Figure S4. Transient Absorption of S-DR1d in hexane at 295.3 K. (blue: 0.16 microseconds, yellow: 3 microseconds, orange: 7.4 microseconds, gray: 15 microseconds)

3. Temperature dependency of the lifetime of S-DR1a-d.

Temperature dependency of the lifetime of S-**DR1a-d** in hexane, benzene, toluene, cyclohexane, methylcyclohexane, acetonitrile, ethyl acetate, 1,4-dioxane, glycerol triacetate (GTA) and 2,4-dicyclohexyl-2-methyl pentane (DCMP) were measured at 5 temperature between 283 K to 323 K under N₂ atmosphere. Temperature dependency of the lifetime of S-**DR1a-d** in pentane were measured at 5 temperature between 263K to 303 K under N₂ atmosphere. Measurements were conducted 5 times at each temperature. The decay profile were monitored at 560 nm(S-**DR1a**), 580 nm(S-**DR1b, c**), and 600 nm (S-**DR1d**).





Figure S5. Decay profile of (a)S-DR1a, (b)1b, (c)1c and (d)1d in pentane at each temperature and (e)Eyring plot of ring closing reaction of S-DR1a-d in pentane.





Figure S6. Decay profile of (a)S-DR1a, (b)1b, (c)1c and (d)1d in hexane at each temperature and (e)Eyring plot of ring closing reaction of S-DR1a-d in hexane.





Figure S7. Decay profile of (a)S-DR1a, (b)1b, (c)1c and (d)1d in benzene at each temperature and (e)Eyring plot of ring closing reaction of S-DR1a-d in benzene.





Figure S8. Decay profile of (a)S-DR1a, (b)1b, (c)1c and (d)1d in toluene at each temperature and (e)Eyring plot of ring closing reaction of S-DR1a-d in toluene.





Figure S9. Decay profile of (a)S-DR1a, (b)1b, (c)1c and (d)1d in cyclohexane at each temperature and (e)Eyring plot of ring closing reaction of S-DR1a-d in cyclohexane.









Figure S10. Decay profile of (a)S-DR1a, (b)1b, (c)1c and (d)1d in methyl cyclohexane at each temperature and (e)Eyring plot of ring closing reaction of S-DR1a-d in methyl cyclohexane.





Figure S11. Decay profile of (a)S-DR1a, (b)1b, (c)1c and (d)1d in acetonitrile at each temperature and (e)Eyring plot of ring closing reaction of S-DR1a-d in acetonitrile.









Figure S12. Decay profile of (a)S-DR1a, (b)1b, (c)1c and (d)1d in ethyl acetate at each temperature and (e)Eyring plot of ring closing reaction of S-DR1a-d in ethyl acetate.







Figure S13. Decay profile of (a)S-DR1a, (b)1b, (c)1c and (d)1d in 1,4-dioxane at each temperature and (e)Eyring plot of ring closing reaction of S-DR1a-d in 1,4-dioxane.









Figure S14. Decay profile of (a)S-DR1a, (b)1b, (c)1c and (d)1d in GTA at each temperature and (e)Eyring plot of ring closing reaction of S-DR1a-d in GTA.









Figure S15. Decay profile of (a)S-DR1b (b)1c and (c)1d in DCMP at each temperature and (d)Eyring plot of ring closing reaction of S-DR1b-d in DCMP.

4. The lifetime of S-DR1a-d at 293 K and activation parameter for ring closing reaction of S-DR1a-d.

The lifetime τ_{293} of S-**DR1a-d** at 293 K, the activation enthalpy ΔH^{\dagger} and activation entropy ΔS^{\dagger} were determined from the Eyring plot (Figure S5-S15) by regression analysis.

Solvent	π*	$E_{T}(30)/$ kcal mol ⁻¹	η /mPa s ⁻¹	τ ₂₉₃ / μs ª	ΔH^{\ddagger} /kJ mol ^{-1 b}	ΔS^{\ddagger} /kJ mol ⁻¹ T ^{-1b}
Pentane	-0.15	31.0	0.24	0.30±0.04	32.2±0.3	-10.3±1.1
Hexane	-0.11	31.0	0.31	0.31±0.04	30.5±0.3	-15.9±1.1
Benzene	0.55	34.3	0.65	0.53±0.12	32.6±0.5	-13.2±1.7
Toluene	0.49	33.9	0.59	0.52±0.2	34.6±0.8	-6.2±2.5
Cyclohexane	0	30.9	0.98	0.31±0.10	28.8±0.7	-21.7±2.3
Methyl Cyclohexane	-0.01	32.2	0.73	0.35±0.08	30.3±0.5	-17.7±1.7
Acetonitrile	0.66	45.6	0.37	0.63±0.19	30.8±0.6	-21.0±2.1
Ethyl Acetate	0.45	38.1	0.44	0.49±0.11	30.9±0.5	-18.4±1.7
1,4-dioxane	0.49	36.0	1.18	0.59±0.09	34.5±0.4	-7.4±1.2
GTA	0.63	43.3	23	1.12±0.5	38.5±0.9	0.68±3.1

Table S1. Solvent parameters, lifetime of S-DR1a, and activation parameters of ring closing reaction from S-DR1a in each solvent.

[a] τ_{293} were estimated from Eyring plot. The mean error obtained from regression analysis is shown as errors. [b] ΔH^{\ddagger} and ΔS^{\ddagger} were calculated from Eyring plot. The mean error obtained from regression analysis is shown as errors.

Table S2. Solvent parameters, lifetime of S-DR1b, and activation	parameters of ring closing reaction from S-DR1b in each solvent
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Solvent	π*	E _T (30) /kcal mol ⁻¹	η /mPa s ⁻¹	τ ₂₉₃ /μs ª	ΔH^{\ddagger} /kJ mol ^{-1 b}	ΔS^{\ddagger} /kJ mol ⁻¹ T ^{-1 b}
Pentane	-0.15	31.0	0.24	0.83±0.12	32.4±0.3	-17.8±1.2
Hexane	-0.11	31.0	0.31	0.90±0.14	33.4±0.4	-15.0±1.2
Benzene	0.55	34.3	0.65	1.30±0.23	32.7±0.4	-20.3±1.3
Toluene	0.49	33.9	0.59	1.34±0.09	35.4±0.2	-11.6±0.5
Cyclohexane	0	30.9	0.98	1.19±0.17	33.9±0.3	-15.5±1.1
Methyl Cyclohexane	-0.01	32.2	0.73	1.19±0.21	34.2±0.4	-14.8±1.3
Acetonitrile	0.66	45.6	0.37	1.04±0.14	34.1±0.3	-13.7±1.0
Ethyl Acetate	0.45	38.1	0.44	0.97±0.14	34.6±0.3	-11.5±1.1
1,4-dioxane	0.49	36.0	1.18	1.49±0.21	36.9±0.3	-7.0±1.1
GTA	0.63	43.3	23	5.53±1.0	55.6±0.4	45.6±1.4
DCMP	0.14	30.1	38.7	6.19±1.9	57.2±0.7	50.3±2.2

[a] τ_{293} were estimated from Eyring plot. The mean error obtained from regression analysis is shown as errors. [b] ΔH^{\dagger} and ΔS^{\dagger} were calculated from Eyring plot. The mean error obtained from regression analysis is shown as errors.

Table S3. Solvent parameters, lifetime of S-DR1c, and activation parameters of ring closing reaction from S-DR1c in each solvent.

Solvent	π*	E _T (30)/ kcal mol ⁻¹	η /mPa s ⁻¹	τ ₂₉₃ / μs ^a	ΔH^{\ddagger} /kJ mol ^{-1 b}	ΔS^{\ddagger} /kJ mol ⁻¹ T ^{-1 b}
Pentane	-0.15	31.0	0.24	2.32±0.2	34.4±0.2	-19.3±0.6
Hexane	-0.11	31.0	0.31	2.61±0.4	35.0±0.3	-18.4±1.1
Benzene	0.55	34.3	0.65	5.68±0.4	40.6±0.2	-5.6±0.5
Toluene	0.49	33.9	0.59	4.65±0.4	39.4±0.2	-8.0±0.7
Cyclohexane	0	30.9	0.98	8.12±0.6	41.6±0.2	-5.4±0.6
Methyl Cyclohexane	-0.01	32.2	0.73	6.76±0.8	38.8±0.3	-13.4±0.9
Acetonitrile	0.66	45.6	0.37	3.70±1.2	35.9±0.7	-18.1±2.4
Ethyl Acetate	0.45	38.1	0.44	3.69±0.5	39.9±0.3	-4.5±1.0
1,4-dioxane	0.49	36.0	1.18	9.39±0.9	41.7±0.2	-6.0±0.8
GTA	0.63	43.3	23	44.8±16	64.7±0.7	59.4±2.5
DCMP	0.14	30.1	38.7	127±40	66.0±0.7	55.1±2.2

[a] τ_{293} were estimated from Eyring plot. The mean error obtained from regression analysis is shown as errors. [b] ΔH^{\ddagger} and ΔS^{\ddagger} were calculated from Eyring plot. The mean error obtained from regression analysis is shown as errors.

Table S4. Solvent parameters, lifetime of S-DR1d	I, and activation parameters of	ring closing reaction from	S-DR1d in each solvent.
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Solvent []]	π*	E _T (30)/ kcal mol ⁻¹	η /mPa s ⁻¹	τ ₂₉₃ / μs ª	ΔH^{\ddagger} /kJ mol ^{-1 b}	ΔS^{\ddagger} /kJ mol ⁻¹ T ^{-1 b}
Pentane	-0.15	31.0	0.24	5.68±0.4	39.2±0.2	-10.4±0.6
Hexane	-0.11	31.0	0.31	6.59±1.0	41.1±0.3	-5.1±1.1
Benzene	0.55	34.3	0.65	23.8±1.4	45.0±0.1	-2.5±0.5
Toluene	0.49	33.9	0.59	14.6±1.1	43.3±0.2	-4.5±0.6
Cyclohexane	0	30.9	0.98	38.8±4.5	49.4±0.3	8.2±0.9
Methyl Cyclohexane	-0.01	32.2	0.73	24.4±6.8	50.3±0.6	15.2±2.0
Acetonitrile	0.66	45.6	0.37	8.38±1.4	39.8±0.4	-11.8±1.3
Ethyl Acetate	0.45	38.1	0.44	7.89±1.4	44.5±0.4	4.97±1.3
1,4-dioxane	0.49	36.0	1.18	33.3±4.8	50.9±0.3	14.7±1.1
GTA	0.63	43.3	23	77.2±31.5	67.2±0.8	63.4±2.8
DCMP	0.14	30.1	38.7	289±37	70.7±0.3	64.4±1.0

[a] τ_{293} were estimated from Eyring plot. The mean error obtained from regression analysis is shown as errors. [b] ΔH^{\ddagger} and ΔS^{\ddagger} were calculated from Eyring plot. The mean error obtained from regression analysis is shown as errors.

5. The dependency of the lifetime of S-DR1a-d on the solvent polarity

The logarithm of the lifetime τ_{293} of S-**DR1a** in each solvent except GTA were plotted against to π^* and $E_T(30)$ (Figure S16,S17). Both show the linear dependency but coefficient of determination R² for Figure S16, $\ln\tau_{293}$ vs π^* , was better than that for Figure S17, $\ln\tau_{293}$ vs $E_T(30)$. And if we include the lifetime of S-**DR1a** in GTA, the longer-lifetime than the prediction, black dot line, was observed and R² value decreased. These observations were also support that the lifetime of S-**DR1a** were also affected by the solvent viscosity.



Figure S16. Solvent polarity (π^*) dependency of the lifetime of S-DR1a. Black: all solvent in Table S1 except GTA, R² = 0.9594, Blue: all solvent in Table S1, R² = 0.7865.



Figure S17. Solvent polarity (E_T(30)) dependency of the lifetime of S-DR1a. Black: all solvent in Table S1 except GTA, R² = 0.6275, Blue: all solvent in Table S1, R² = 0.5794.

The logarithm of the lifetime of S-**DR1b-d** in all solvent in Table S2-4 were also plotted against the solvent polarity, π^* (Figure S18). But linear relationship was not observed for S-**DR1b-d** cases.



Figure S18. The lifetime of (a) S-DR1b, (b)S-DR1c and (c)S-DR1d dependency on the solvent polarity: π*. Solvent: Benzene, Hexane, Acetonitrile, Cyclohexane, Toluene, Pentane, 1,4dioxane, Ethyl Acetate, Methyl Cyclohexane, GTA, DCMP.

6. Determination of the reaction path and photoproduct of S-DR1a-d under high viscous condition

6.1 NMR analysis of photo-product of S-DR1d in DCMP

To clarify the reaction path and product of S-**DR1d** under high viscous condition, we compare the product in DCMP and deuterium benzene. **AZ1d** (3 mg, 3 mmol) were dissolved in degassed DCMP (0.3 ml). This solution was irradiated by the 365 nm LED lamp, and the reaction was monitored by ¹H-NMR. During the photoirradiation, the signals around 7.7 and 5.4 ppm decreased, and the signals around 6.9, 6.7, and 5.7 ppm newly appeared (Figure S19). Compared with the **CP1d** in DCMP, which were prepared from denitrogenation of **AZ1d** in C₆D₆, these newly observed peaks in photoirradiation of **AZ1d** in DCMP were matched with **CP1d**. So, we conclude the reaction path, which corresponds to the lifetime of S-**DR1d**, was the bond formation process of the radicals. These ¹H NMR spectra were measured on a Bruker ASCENDTM 400 spectrometer.



Figure S19. ¹H NMR (400 MHz) of AZ1d(bottom), during photoirradiation with LED lamp(365 nm) (middle) and CP1d (top) in DCMP. C₆D₆ were used for external standard for lock.

6.2. Enthalpy-Entropy Compensation for ring-closing reaction of S-DR1a-d

Enthalpy-entropy compensation for ring-closing reaction in each solvent suggested that the transition state was not changed in all solvents (Figure S20). And previously it was confirmed that singlet diradicals S-DR1a-d went to ring-closure product CP1a-d in benzene. So, these results also support that all singlet diradicals S-DR1a-d.



Figure S20. Enthalpy entropy compensation graph. (Blue: S-DR1a; slope = 0.3771, R^2 = 0.9194, Orange: S-DR1b; slope = 0.3575, R^2 = 0.998, gray: S-DR1c; slope = 0.3924, R^2 = 0.9914, yellow: S-DR1d; slope = 0.3881, R^2 = 0.984.)

7. High-pressure experiments

7.1 S-DR1d in DCMP



Figure S21. Time profile of S-DR1d in DCMP under 1000 atm at 293 K. (τ = 4.65 ms)



Figure S22. Time profile of S-DR1d in DCMP under 2000 atm at 293 K. (τ = 53.9 ms)



Figure S23. Time profile of S-DR1d in DCMP under 3000 atm at 293 K. (τ = 352 ms)



7.2 S-DR1d in GTA

Figure S24. Time profile of S-DR1d in GTA under 1000 atm at 293 K. (τ = 317 microseconds)



Figure S25. Time profile of S-DR1d in GTA under 2000 atm at 293 K. (τ = 1280 microseconds = 1.28 ms)



Figure S26. Time profile of S-DR1d in GTA under 3000 atm at 293 K. (τ = 20636 microseconds = 20.6 ms)



Figure S27. Time profile of S-DR1d in GTA under 4000 atm at 293 K. (τ = 138 ms)



Figure S28. Time profile of S-DR1d in GTA under 5000 atm at 293 K. (τ = 371 ms)



Figure S29. Time profile of S-DR1d in GTA under 6000 atm at 293 K. (τ = 727 ms)



7.3 S-DR1d in MCH

Figure S30. Time profile of S-DR1d in MCH under 250 atm at 293 K. (τ = 34.0 microseconds)



Figure S31. Time profile of S-DR1d in MCH under 500 atm at 293 K. (τ = 38.3 microseconds)



Figure S32. Time profile of S-DR1d in MCH under 750 atm at 293 K. (τ = 43.8 microseconds)



Figure S33. Time profile of S-DR1d in MCH under 1000 atm at 293 K. (τ = 59.5 microseconds)



Figure S34. Time profile of S-DR1d in MCH under 1250 atm at 293 K. (τ = 75.4 microseconds)



Figure S35. Time profile of S-DR1a in GTA under 250 atm at 293 K. (τ = 1.25 microseconds)



Figure S36. Time profile of S-DR1a in GTA under 535 atm at 293 K. (τ = 1.69 microseconds)



Figure S37. Time profile of S-DR1a in GTA under 750 atm at 293 K. (τ = 2.10 microseconds)



Figure S38. Time profile of S-DR1a in GTA under 1000 atm at 293 K. (τ = 2.61 microseconds)



Figure S39. Time profile of S-DR1a in GTA under 2000 atm at 293 K. (τ = 10.8 microseconds)



Figure S40. Time profile of S-DR1a in GTA under 3000 atm at 293 K. (τ = 58.3 microseconds)



Figure S41. Time profile of S-DR1a in GTA under 4000 atm at 293 K. (τ = 380 microseconds)



Figure S42. Time profile of S-DR1a in GTA under 5000 atm at 293 K. (τ = 3570 microseconds = 3.57 ms)

7.4 S-DR1a in MCH



Figure S43. Time profile of S-DR1a in MCH under 250 atm at 293 K. (τ = 445 ns)



Figure S44. Time profile of S-DR1a in MCH under 500 atm at 293 K. (τ = 449 ns)



Figure S45. Time profile of S-DR1a in MCH under 750 atm at 293 K. (τ = 544 ns)



Figure S46. Time profile of S-DR1a in MCH under 1000 atm at 293 K. (τ = 542 ns)



Figure S47. Time profile of S-DR1a in MCH under 1250 atm at 293 K. (τ = 459 ns)



Figure S48. Time profile of S-DR1a in MCH under 1500 atm at 293 K. (τ = 466 ns)



Figure S49. Time profile of S-DR1a in MCH under 1750 atm at 293 K. (τ = 628 ns)



Figure S50. Time profile of S-DR1a in MCH under 2000 atm at 293 K. (τ = 504 ns)



Figure S51. Time profile of S-DR1a in MCH under 3000 atm at 293 K. (τ = 600 ns)



Figure S52. Time profile of S-DR1a in MCH under 4000 atm at 293 K. (τ = 814 ns)

8. Determination of solvent polarity parameter, π^*

The UV-vis absorption spectra of 4-nitro-anisole were measured in MCH and DCMP. From the wavelength of absorption maximum, λ_{max} , π^* values of each solvent were calculated from the literature equation, $10^4/\lambda_{max} = 34.17 - 2.41 \text{ p}^*$. ^[4] These UV-vis absorption measurements were recorded on a SHIMADZU UV-3600 Plus spectrometer.



Figure S53. UV-vis absorption spectra of 4-nitro-anisole in MCH (Blue), and DCMP(Orange).

Table S5. Determination of p* for MCH and DCMP

Solvent	$\lambda_{max}/nm^{[a]}$	π*
МСН	292.55	-0.01
DCMP	295.55	0.14

[a] Absorption maximum λ_{max} of 4-nitro-anisole in each solvents.

9. Calculation of the pressure dependency of solvent viscosity for methyl cyclohexane

The pressure dependency of the solvent viscosity for methyl cyclohexane at 293 K were examined by Zéberg-Mikkelsen *et. al.*^[7] And these data were fitted with equation, $\ln \eta = a P + \ln \eta_0$, by regression analysis (Figure S54). From these parameters, α and η_0 , solvent viscosity for each pressure were calculated (Table S6).



Figure S54. Pressure dependency of the viscosity for Methyl cyclohexane.

Table S6.	Estimated	Viscosity fo	r methyl c	vclohexane ;	at each Pressure
Table 50.	Lounducu	viscosity io	i incuityi c	ycionic and a	

Pressure /atm	1	250	500	750	1000	1250
In (η /mPa s-1)	-0.2707	-0.0359	0.1999	0.43566	0.67142	0.90718
η /mPa s ⁻¹	0.763	0.965	1.22	1.54	1.96	2.48

10. References

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