Supporting Information

K₂S₂O₈-Mediated Difunctionalization of C≡C Bonds in Water: a Simple and Efficient

Approach to α,α -Dihaloacetophenones from Phenylacetylenes and NaX

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General Consideration

All reagents and solvent were purchased commercially and used without further purification.

Mass spectra were measured on a mass instrument (EI). ^1H NMR spectra were recorded on 400 MHz in CDCl_3 , and ^{13}C NMR spectra were recorded on 101 MHz in CDCl_3 using TMS as internal standard. Multiplicities are indicated as s (singlet), d (doublet), t (triplet), q (quartet), and m (multiplet), and coupling constants (J) are reported in hertz. Copies of ^1H NMR and ^{13}C NMR spectra are provided as Supporting Information.

Table S1. The Screening of the Reaction Conditions^a

Entry	Oxidant (equiv)	Halogen source (equiv)	Solvent	T ($^{\circ}\text{C}$)	Time (h)	Yield (%) ^b
1	$\text{K}_2\text{S}_2\text{O}_8$ (2.5)	NaBr (2.0)	H_2O	60	12	85
2	None	NaBr (2.0)	H_2O	60	12	0
3	$\text{K}_2\text{S}_2\text{O}_8$ (2.5)	KBr (2.0)	H_2O	60	12	68
4	$\text{K}_2\text{S}_2\text{O}_8$ (2.5)	LiBr (2.0)	H_2O	60	12	75
5	$\text{K}_2\text{S}_2\text{O}_8$ (2.5)	NH_4Br (2.0)	H_2O	60	12	70
6	$\text{K}_2\text{S}_2\text{O}_8$ (2.5)	NaBr (1.0)	H_2O	60	12	37
7	$\text{K}_2\text{S}_2\text{O}_8$ (2.5)	NaBr (1.5)	H_2O	60	12	74
8	$\text{K}_2\text{S}_2\text{O}_8$ (2.5)	NaBr (2.5)	H_2O	60	12	67
9	$\text{K}_2\text{S}_2\text{O}_8$ (2.5)	NaBr (3.0)	H_2O	60	12	56
10	$\text{Na}_2\text{S}_2\text{O}_8$ (2.5)	NaBr (2.0)	H_2O	60	12	74
11	$(\text{NH}_4)_2\text{S}_2\text{O}_8$ (2.5)	NaBr (2.0)	H_2O	60	12	68
12	$\text{K}_2\text{S}_2\text{O}_8$ (1.0)	NaBr (2.0)	H_2O	60	12	52
13	$\text{K}_2\text{S}_2\text{O}_8$ (1.5)	NaBr (2.0)	H_2O	60	12	63
14	$\text{K}_2\text{S}_2\text{O}_8$ (2.0)	NaBr (2.0)	H_2O	60	12	76
15	$\text{K}_2\text{S}_2\text{O}_8$ (3.0)	NaBr (2.0)	H_2O	60	12	57
16	$\text{K}_2\text{S}_2\text{O}_8$ (2.5)	NaBr (2.0)	$\text{CH}_3\text{OH}-\text{H}_2\text{O}$ (1/1)	60	12	64
17	$\text{K}_2\text{S}_2\text{O}_8$ (2.5)	NaBr (2.0)	$\text{CH}_3\text{CN}-\text{H}_2\text{O}$ (1/1)	60	12	65
18	$\text{K}_2\text{S}_2\text{O}_8$ (2.5)	NaBr (2.0)	CH_3OH	60	12	0
19	$\text{K}_2\text{S}_2\text{O}_8$ (2.5)	NaBr (2.0)	$\text{CH}_3\text{CH}_2\text{OH}$	60	12	0
20	$\text{K}_2\text{S}_2\text{O}_8$ (2.5)	NaBr (2.0)	CH_3CN	60	12	0
21	$\text{K}_2\text{S}_2\text{O}_8$ (2.5)	NaBr (2.0)	H_2O	50	12	75
22	$\text{K}_2\text{S}_2\text{O}_8$ (2.5)	NaBr (2.0)	H_2O	70	12	68

23	K ₂ S ₂ O ₈ (2.5)	NaBr (2.0)	H ₂ O	RT	12	0
24	K ₂ S ₂ O ₈ (2.5)	NaBr (2.0)	H ₂ O	60	10	73
25	K ₂ S ₂ O ₈ (2.5)	NaBr (2.0)	H ₂ O	60	15	78
26	K ₂ S ₂ O ₈ (2.5)	KCl (2.0)	H ₂ O	60	12	45
27	K ₂ S ₂ O ₈ (2.5)	NaCl (2.0)	H ₂ O	60	12	58
28	K ₂ S ₂ O ₈ (2.5)	LiCl (2.0)	H ₂ O	60	12	49
29	K ₂ S ₂ O ₈ (2.5)	NH ₄ Cl (2.0)	H ₂ O	60	12	42
30	Na ₂ S ₂ O ₈ (2.5)	NaCl (2.0)	H ₂ O	60	12	46
31	(NH ₄) ₂ S ₂ O ₈ (2.5)	NaCl (2.0)	H ₂ O	60	12	36
32	K ₂ S ₂ O ₈ (2.5)	NaCl (1.5)	H ₂ O	60	12	50
33	K ₂ S ₂ O ₈ (2.5)	NaCl (2.5)	H ₂ O	60	12	47
34	K ₂ S ₂ O ₈ (2.0)	NaCl (2.0)	H ₂ O	60	12	48
35	K ₂ S ₂ O ₈ (3.0)	NaCl (2.0)	H ₂ O	60	12	36
36	K ₂ S ₂ O ₈ (2.5)	NaCl (2.0)	CH ₃ OH-H ₂ O (1/1)	60	12	28
37	K ₂ S ₂ O ₈ (2.5)	NaCl (2.0)	CH ₃ CN-H ₂ O (1/1)	60	12	25
38	K ₂ S ₂ O ₈ (2.5)	NaCl (2.0)	CH ₃ OH	60	12	0
39	K ₂ S ₂ O ₈ (2.5)	NaCl (2.0)	CH ₃ CH ₂ OH	60	12	0
40	K ₂ S ₂ O ₈ (2.5)	NaCl (2.0)	H ₂ O	70	12	35
41	K ₂ S ₂ O ₈ (2.5)	NaCl (2.0)	H ₂ O	50	12	44
[a] Conditions: phenylacetylene (0.5 mmol), oxidant, halogen source, solvent (1 mL). [b] Yields are determined by GC.						

Halide salts and persulfate salts were investigated in the model reaction of phenylacetylene. As shown in Table S1, NaX and K₂S₂O₈ turned out to be the best choices, while others such as LiX, KX, NH₄X, Na₂S₂O₈ and (NH₄)₂S₂O₈ were low effective. The influence of the cations on the reaction was not clear at the present stage. BUT, the yield differences obtained using bromide having different cation is experimental fact. To further confirm the fact, the reactions of phenylacetylene with NaBr, KBr and LiBr were repeated five times, and five runs were almost consistent in yields (see below). Further studies on the effect and development of relevant reactions are still currently under investigation.

Table S2. Repeated Experiments using NaBr.

Run	1	2	3	4	5
Yield (%)	85	85	86	84	85

Table S3. Repeated Experiments using KBr.

Run	1	2	3	4	5
Yield (%)	68	67	68	68	68

Table S4. Repeated Experiments using LiBr.

Run	1	2	3	4	5
Yield (%)	75	75	75	74	74

General procedure for the synthesis of α,α -dihaloacetophenones via $K_2S_2O_8$ -mediated oxy-1,1-dihalogenation of alkynes (2a-2m, 3a-3j)

To a 25 mL Schlenck tube were added $K_2S_2O_8$ (2.5 equiv.), NaX (2.0 equiv.), alkynes (0.5 mmol) and H_2O (1 mL). The reaction mixture was warmed to 60 °C (oil bath) and stirred for 12 h. The reaction was cooled to room temperature, and ethyl acetate (5 mL) and water (2 mL) were added. The organic layer was separated, and the aqueous phase was extracted with ethyl acetate (10 mL $\times$ 2). The combined organic layers were dried over anhydrous Na_2SO_4 , filtered, and concentrated. The residue was purified by column chromatography to give desired product.

Scheme S1. The reaction of phenylacetylene with KI in the presence of $K_2S_2O_8$

Figure S1. NMR spectra of the mixture of the reaction of 1-heptyne with NaCl in the presence of $K_2O_2S_8$

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Characterization Data

2,2-Dibromo-1-phenylethanone (**2a**):^[1] Flash chromatography (petroleum ether/ dichloromethane, 7/1); Yield: 78%, pale yellow oil; ¹H NMR (CDCl₃, 400 MHz) δ 6.75 (s, 1 H), 7.50 (t, J = 8.0 Hz, 2 H), 7.63 (t, J = 8.0 Hz, 1 H), 8.07 (d, J = 8.0 Hz, 2 H); ¹³C NMR (CDCl₃, 101 MHz) δ 39.9, 129.0, 129.7, 130.8, 134.5, 186.0; LRMS: m/z calcd for C₈H₆Br₂O (M+H): 279, found: 279.

2,2-Dibromo-1-(4-fluorophenyl)ethanone (**2b**):^[2] Flash chromatography (petroleum ether/ dichloromethane, 7/1); Yield: 75%, pale yellow oil; ¹H NMR (CDCl₃, 400 MHz) δ 6.64 (s, 1 H), 7.19 (t, J = 8.0 Hz, 2 H), 8.15 (t, J = 8.0 Hz, 2 H); ¹³C NMR (CDCl₃, 101 MHz) δ 39.4, 116.2 (d, J_{C-F} = 22.2 Hz), 127.1 (d, J_{C-F} = 3.0 Hz), 132.7 (d, J_{C-F} = 10.1 Hz), 166.3 (d, J_{C-F} = 259.6 Hz), 184.6; LRMS: m/z calcd for C₈H₅Br₂FO (M+H): 297, found: 297.

2,2-Dibromo-1-(4-chlorophenyl)ethanone (**2c**):^[3] Flash chromatography (petroleum ether/ dichloromethane, 5/1); Yield: 64%, pale yellow solid, Mp = 94-96 °C; ¹H NMR (CDCl₃, 400 MHz) δ 6.54 (s, 1 H), 7.42 (d, J = 8.0 Hz, 2 H), 7.98 (d, J = 8.0 Hz, 1 H); ¹³C NMR (CDCl₃, 101 MHz) δ 39.3, 129.1, 129.3, 131.2, 141.1, 184.9; LRMS: m/z calcd for C₈H₅Br₂ClO (M+H): 313, found: 313.

2,2-Dibromo-1-(4-bromophenyl)ethanone (**2d**):^[3] Flash chromatography (petroleum ether/ dichloromethane, 7/1); Yield: 61%, colorless solid, Mp = 92-94 °C; ¹H NMR (CDCl₃, 400 MHz) δ 6.60 (s, 1 H), 7.66 (d, J = 8.0 Hz, 2 H), 7.97 (d, J = 8.0 Hz, 1 H); ¹³C NMR (CDCl₃, 101 MHz) δ 39.2, 129.5, 129.9, 131.2, 132.3, 185.1; LRMS: m/z calcd for C₈H₅Br₃O (M+H): 358, found: 358.

2,2-Dibromo-1-(4-methylphenyl)ethanone (**2e**):^[1] Flash chromatography (petroleum ether/ dichloromethane, 7/1); Yield: 74%, pale yellow solid, Mp = 87-89 °C; ¹H NMR (CDCl₃, 400 MHz) δ 2.44 (s, 3 H), 6.70 (s, 1 H), 7.31 (d, J = 8.0 Hz, 2 H), 7.98 (d, J = 8.0 Hz, 1 H); ¹³C NMR (CDCl₃, 101 MHz) δ 21.9, 39.9, 128.2, 129.7, 129.8, 145.8, 185.7; LRMS: m/z calcd for

$C_9H_8Br_2O$ (M+H): 293, found: 293.

2,2-Dibromo-1-(3-methylphenyl)ethanone (**2f**):^[4] Flash chromatography (petroleum ether/dichloromethane, 7/1); Yield: 72%, pale yellow oil; 1H NMR ($CDCl_3$, 400 MHz) δ 2.44 (s, 3 H), 6.73 (s, 1 H), 7.37-7.46 (m, 2 H), 7.87 (s, 2 H); ^{13}C NMR ($CDCl_3$, 101 MHz) δ 21.4, 39.9, 126.8, 128.8, 130.1, 130.9, 135.3, 139.0, 186.2; LRMS: m/z calcd for $C_9H_8Br_2O$ (M+H): 293, found: 293.

2,2-Dibromo-1-(4-ethylphenyl)ethanone (**2g**):^[5] Flash chromatography (petroleum ether/dichloromethane, 7/1); Yield: 67%, pale yellow oil; 1H NMR ($CDCl_3$, 400 MHz) δ 1.20 (t, 3 H), 2.67 (q, $J = 8.0$ Hz, 2 H), 6.63 (s, 1 H), 7.26 (d, $J = 8.0$ Hz, 2 H), 7.94 (d, $J = 8.0$ Hz, 2 H); ^{13}C NMR ($CDCl_3$, 101 MHz) δ 15.0, 29.1, 39.9, 128.4, 128.5, 130.0, 151.9, 185.7; LRMS: m/z calcd for $C_{10}H_{10}Br_2O$ (M+H): 307, found: 307.

2,2-Dibromo-1-(4-propylphenyl)ethanone (**2h**): Flash chromatography (petroleum ether/dichloromethane, 7/1); Yield: 62%, pale yellow oil; 1H NMR ($CDCl_3$, 400 MHz) δ 0.88 (t, $J = 8.0$ Hz, 3 H), 1.56-1.65 (m, 2 H), 2.59 (t, $J = 8.0$ Hz, 2 H), 6.63 (s, 1 H), 7.23 (d, $J = 8.0$ Hz, 2 H), 7.93 (d, $J = 8.0$ Hz, 2 H); ^{13}C NMR ($CDCl_3$, 101 MHz) δ 13.1, 24.1, 38.2, 39.9, 128.4, 129.1, 129.9, 150.4, 185.7; LRMS: m/z calcd for $C_{11}H_{12}Br_2O$ (M+H): 321 found: 321.

2,2-Dibromo-1-(4-butylphenyl)ethanone (**2i**):^[6] Flash chromatography (petroleum ether/dichloromethane, 7/1); Yield: 64%, pale yellow oil; 1H NMR ($CDCl_3$, 400 MHz) δ 0.93 (t, $J = 8.0$ Hz, 3 H), 1.31-1.41 (m, 2 H), 1.58-1.66 (m, 2H), 2.68 (t, $J = 8.0$ Hz, 2 H), 6.73 (s, 1 H), 7.30 (d, $J = 8.0$ Hz, 2 H), 7.99 (d, $J = 8.0$ Hz, 2 H); ^{13}C NMR ($CDCl_3$, 101 MHz) δ 14.0, 22.4, 33.1, 35.8, 40.1, 128.4, 129.1, 129.9, 150.6, 185.7; LRMS: m/z calcd for $C_{12}H_{14}Br_2O$ (M+H): 335, found: 335.

2,2-Dibromo-1-(4-amyphenyl)ethanone (**2j**):^[7] Flash chromatography (petroleum ether/dichloromethane, 7/1); Yield: 65%, colorless oil; ¹H NMR (CDCl₃, 400 MHz) δ 0.89 (t, J = 8.0 Hz, 3 H), 1.26-1.37 (m, 4 H), 1.60-1.68 (m, 2H), 2.68 (t, J = 8.0 Hz, 2 H), 6.74 (s, 1 H), 7.31 (d, J = 8.0 Hz, 2 H), 8.00 (d, J = 8.0 Hz, 2 H); ¹³C NMR (CDCl₃, 101 MHz) δ 14.0, 22.5, 30.7, 31.4, 36.1, 40.0, 128.4, 129.0, 129.9, 150.7, 185.7; LRMS: m/z calcd for C₁₃H₁₆Br₂O (M+H): 349, found: 349.

2,2-Dibromo-1-phenylpropan-1-one (**2k**):^[1] Flash chromatography (petroleum ether); Yield: 55%, colorless yellow oil; ¹H NMR (CDCl₃, 400 MHz) δ 2.75 (s, 3 H), 7.47 (t, J = 8.0 Hz, 2 H), 7.58 (t, J = 8.0 Hz, 1 H), 8.40 (d, J = 8.0 Hz, 2 H); ¹³C NMR (CDCl₃, 101 MHz) δ 37.7, 57.9, 128.0, 131.4, 131.7, 133.5, 188.3; LRMS: m/z calcd for C₉H₈Br₂O (M+H): 293, found: 293.

2,2-Dibromo-1-phenylbutan-1-one (**2l**):^[6] Flash chromatography (petroleum ether); Yield: 52%, colorless oil; ¹H NMR (CDCl₃, 400 MHz) δ 1.29 (t, J = 8.0 Hz, 3 H), 2.71 (q, J = 8.0 Hz, 2 H), 7.46 (t, J = 8.0 Hz, 2 H), 7.57 (t, J = 8.0 Hz, 1 H), 8.35 (d, J = 8.0 Hz, 2 H); ¹³C NMR (CDCl₃, 101 MHz) δ 11.7, 40.2, 68.4, 128.1, 131.1, 132.6, 133.4, 188.7; LRMS: m/z calcd for C₁₀H₁₀Br₂O (M+H): 307, found: 307.

(*E*)-1,2-Dibromohept-1-ene (**2m**):^[8] Flash chromatography (hexane); Yield: 58%, colorless oil; ¹H NMR (CDCl₃, 400 MHz) δ 0.92 (t, J = 8.0 Hz, 3 H), 1.34 (m, 4 H), 1.57-1.62 (m, 2 H), 2.59 (t, J = 8.0 Hz, 2 H), 6.40 (s, 1 H); ¹³C NMR (CDCl₃, 101 MHz) δ 14.0, 22.4, 26.7, 30.5, 36.9, 102.1, 127.1; LRMS: m/z calcd for C₇H₁₂Br₂ (M+H): 257, found: 257.

2,2-Dichloro-1-phenylethanone (**3a**):^[7] Flash chromatography (petroleum ether/ dichloromethane, 7/1); Yield: 52%, colorless oil; ¹H NMR (CDCl₃, 400 MHz) δ 6.69 (s, 1 H), 7.53 (t, J = 8.0 Hz, 2 H), 7.66 (t, J = 8.0 Hz, 1 H), 8.09 (d, J = 8.0 Hz, 2 H); ¹³C NMR (CDCl₃, 101 MHz) δ 67.8, 128.9,

129.8, 131.3, 134.6, 185.9; LRMS: m/z calcd for $C_8H_6Cl_2O$ (M+H): 190, found: 190.

2,2-Dichloro-1-(4-fluorophenyl)ethanone (**3b**):^[9] Flash chromatography (petroleum ether/dichloromethane, 7/1); Yield: 49%, pale yellow oil; 1H NMR ($CDCl_3$, 400 MHz) δ 6.60 (s, 1 H), 7.20 (t, J = 8.0 Hz, 2 H), 8.14-8.17 (m, 2 H); ^{13}C NMR ($CDCl_3$, 101 MHz) δ 67.82, 116.2 (d, J_{C-F} = 22.2 Hz), 127.5 (d, J_{C-F} = 3.0 Hz), 132.8 (d, J_{C-F} = 10.1 Hz), 166.4 (d, J_{C-F} = 258.6 Hz), 184.6; LRMS: m/z calcd for $C_8H_5Cl_2FO$ (M+H): 208, found: 208.

2,2-Dichloro-1-(4-chlorophenyl)ethanone (**3c**):^[9] Flash chromatography (petroleum ether/dichloromethane, 7/1); Yield: 38%, pale yellow solid, Mp = 56-58 °C;^[9] 1H NMR ($CDCl_3$, 400 MHz) δ 6.52 (s, 1 H), 7.43 (d, J = 12.0 Hz, 2 H), 7.99 (d, J = 8.0 Hz, 2 H); ^{13}C NMR ($CDCl_3$, 101 MHz) δ 67.8, 129.3, 129.5, 131.3, 141.3, 185.0; LRMS: m/z calcd for $C_8H_5Cl_3O$ (M+H): 224, found: 224.

2,2-Dichloro-1-(4-bromophenyl)ethanone (**3d**):^[10] Flash chromatography (petroleum ether/dichloromethane, 7/1); Yield: 35%, colorless solid, Mp = 50-52 °C;^[10] 1H NMR ($CDCl_3$, 400 MHz) δ 6.51 (s, 1 H), 7.60 (d, J = 8.0 Hz, 2 H), 7.90 (d, J = 8.0 Hz, 2 H); ^{13}C NMR ($CDCl_3$, 101 MHz) δ 67.8, 130.1, 131.2, 131.3, 132.3, 185.2; LRMS: m/z calcd for $C_8H_5Cl_2BrO$ (M+H): 269, found: 269.

2,2-Dichloro-1-(4-methylphenyl)ethanone (**3e**):^[10] Flash chromatography (petroleum ether/dichloromethane, 7/1); Yield: 46%, pale yellow solid, m.p. = 52-54 °C;^[10] 1H NMR ($CDCl_3$, 400 MHz) δ 2.45 (s, 3 H), 6.67 (s, 1 H), 7.32 (d, J = 8.0 Hz, 2 H), 7.99 (d, J = 8.0 Hz, 2 H); ^{13}C NMR ($CDCl_3$, 101 MHz) δ 21.9, 67.8, 128.7, 129.7, 129.9, 145.9, 185.6; LRMS: m/z calcd for $C_9H_8Cl_2O$ (M+H): 204, found: 204.

2,2-Dichloro-1-(3-methylphenyl)ethanone (**3f**): Flash chromatography (petroleum ether/

dichloromethane, 7/1); Yield: 43%, pale yellow oil; ^1H NMR (CDCl_3 , 400 MHz) δ 2.37 (s, 3 H), 6.63 (s, 1 H), 7.32-7.41 (m, 2 H), 7.81 (s, 2 H); ^{13}C NMR (CDCl_3 , 101 MHz) δ 20.4, 66.7, 125.8, 127.7, 129.1, 130.4, 134.4, 137.9, 185.0; LRMS: m/z calcd for $\text{C}_9\text{H}_8\text{Cl}_2\text{O}$ (M+H): 204, found: 204.

2,2-Dichloro-1-(4-ethylphenyl)ethanone (**3g**):^[11] Flash chromatography (petroleum ether/dichloromethane, 7/1); Yield: 40%, pale yellow oil; ^1H NMR (CDCl_3 , 400 MHz) δ 1.28 (t, $J = 8.0$ Hz, 3 H), 2.74 (q, $J = 8.0$ Hz, 2 H), 6.68 (s, 1 H), 7.35 (d, $J = 8.0$ Hz, 2 H), 8.01 (d, $J = 8.0$ Hz, 2 H); ^{13}C NMR (CDCl_3 , 101 MHz) δ 15.0, 29.0, 67.8, 128.5, 128.9, 130.0, 152.0, 185.6; LRMS: m/z calcd for $\text{C}_{10}\text{H}_{10}\text{Cl}_2\text{O}$ (M+H): 218, found: 218.

2,2-Dichloro-1-phenylpropan-1-one (**3h**):^[9] Flash chromatography (hexane); Yield: 37%, pale yellow oil; ^1H NMR (CDCl_3 , 400 MHz) δ 2.36 (s, 3 H), 7.48 (t, $J = 8.0$ Hz, 2 H), 7.59 (t, $J = 8.0$ Hz, 1 H), 8.32 (d, $J = 8.0$ Hz, 2 H); ^{13}C NMR (CDCl_3 , 101 MHz) δ 34.3, 82.7, 128.1, 131.2, 131.27, 133.6, 188.1; LRMS: m/z calcd for $\text{C}_9\text{H}_8\text{Cl}_2\text{O}$ (M+H): 204, found: 204.

2,2-Dichloro-1-phenylbutan-1-one (**3i**):^[12] Flash chromatography (hexane); Yield: 34%, colorless oil; ^1H NMR (CDCl_3 , 400 MHz) δ 1.26 (t, $J = 8.0$ Hz, 3 H), 2.54 (q, $J = 8.0$ Hz, 2 H), 7.47 (t, $J = 8.0$ Hz, 2 H), 7.59 (t, $J = 8.0$ Hz, 1 H), 8.28 (d, $J = 8.0$ Hz, 2 H); ^{13}C NMR (CDCl_3 , 101 MHz) δ 9.2, 37.8, 88.4, 128.1, 131.0, 131.5, 133.4, 188.5; LRMS: m/z calcd for $\text{C}_{10}\text{H}_{10}\text{Cl}_2\text{O}$ (M+H): 218, found: 218.

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NMR Spectra

NMR spectra for **2a**

NMR spectra for **2b**

NMR spectra for **2c**

NMR spectra for **2d**

NMR spectra for **2e**

NMR spectra for **2f**

NMR spectra for **2g**

NMR spectra for **2h**

NMR spectra for **2i**

NMR spectra for **2j**

NMR spectra for **2k**

NMR spectra for **21**

NMR spectra for **2m**

NMR spectra for **3a**

NMR spectra for **3b**

NMR spectra for **3c**

NMR spectra for **3d**

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NMR spectra for **3e**

NMR spectra for **3f**

NMR spectra for **3h**

NMR spectra for **3i**