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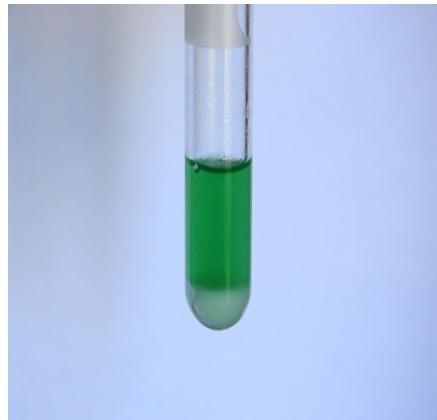
General experimental details

All reagents were obtained from commercial sources. All reactions were run under atmospheric conditions unless otherwise indicated. Yields reported are for isolated yields unless otherwise noted. $[\text{Rh}_2(\text{cap})_4 \cdot 2\text{CH}_3\text{CN}]$ was purchased from Alfa Aesar. $\text{Rh}_2(\text{esp})_2$ was purchased from Aldrich or prepared as described.¹ The preparation of $\text{Rh}_2(\text{MPDP})_2$ and $\text{Rh}_2(\text{OAc})_4(\text{IMes})$ have been previously described.^{2,4} $\text{Rh}_2(\text{MPDP})(\text{CO}_2\text{CF}_3)_2$ was prepared as Taber and co-workers described.³ *tert*-Butyl hydroperoxide (TBHP) was purchased from Aldrich as a 5.5 M solution in decane and stored over activated 3Å molecular sieves. 70% *tert*-Butyl hydroperoxide in water (T-HYDRO) was purchased from Alfa Aesar. Gas Chromatography (GC) spectra were obtained on Agilent 7820A using DB-624 column. UV/visible spectroscopy spectra were carried out on Shimadzu UV-2450. ¹H NMR and ¹³NMR spectra were measured on a 300 MHz or 400 MHz Bruker spectrometer using CDCl_3 as the solvent. Chemical shifts are given in δ relative to TMS. High resolution mass spectrometric measurements were carried out using a Bruker autoflex MALDI-TOF mass spectrometer and Waters-Q-TOF Premier (ESI).

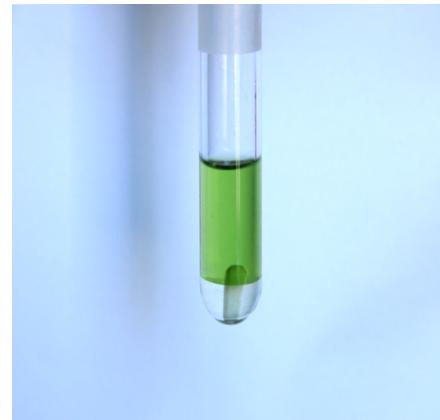
General Procedures for Products

A 10 mL tube equipped with a stir bar was charged with substrate (4 mmol) and $\text{Rh}_2(\text{esp})_2$ (3 mg, 0.004 mmol), then sealed by a rubber plug with a needle, followed by the addition of T-HYDRO (2.84 mL, 20 mmol) via syringe. After 24 hours, saturated sodium thiosulfate solution was added to quench the reaction. The mixture was extracted with ethyl acetate (3×5 mL) and washed with water (2×10 mL). The ethyl acetate layer was separated and dried over MgSO_4 . After evaporation of the solvent, the residue was purified by flash column chromatography (ethyl acetate/hexane) to give the desired products.

The Pictures of the Oxidation Reaction of $\square\alpha$ -Isophorone Catalyzed by $\text{Rh}_2(\text{esp})_2$ and $\text{Rh}_2(\text{cap})_4$

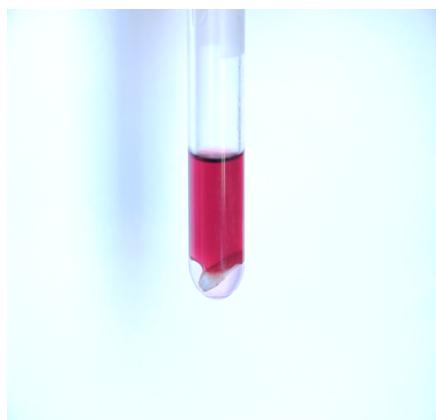


at the beginning of the reaction.

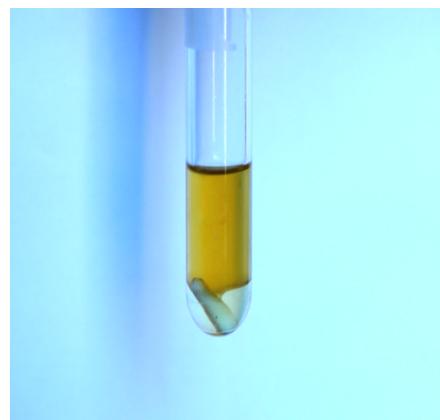


at the end of the reaction

Figure 1 the reaction catalyzed by $\text{Rh}_2(\text{esp})_2$



at the beginning of the reaction



at the end of the reaction

Figure 2 the reaction catalyzed by $\text{Rh}_2(\text{cap})_4$

Gas Chromatography (GC) analysis of oxidation of $\square\alpha$ -Isophorone catalyzed by $\text{Rh}_2(\text{esp})_2$ and $\text{Rh}_2(\text{OAc})_4$

Gas Chromatography (GC) spectra were obtained on Agilent 7820A using DB-624 column (30 m × 0.32 mm). GC conditions: isotherm at 40 °C (6 min); ramp at 10 °C min⁻¹ to 180 °C; isotherm at 180 °C (5 min).

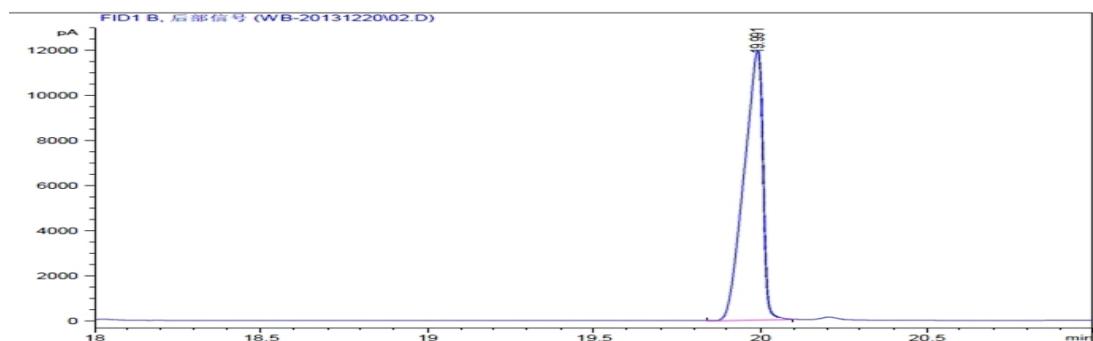


Figure 3 **α-1** (α -Isophorone) $t_R = 19.99$ min

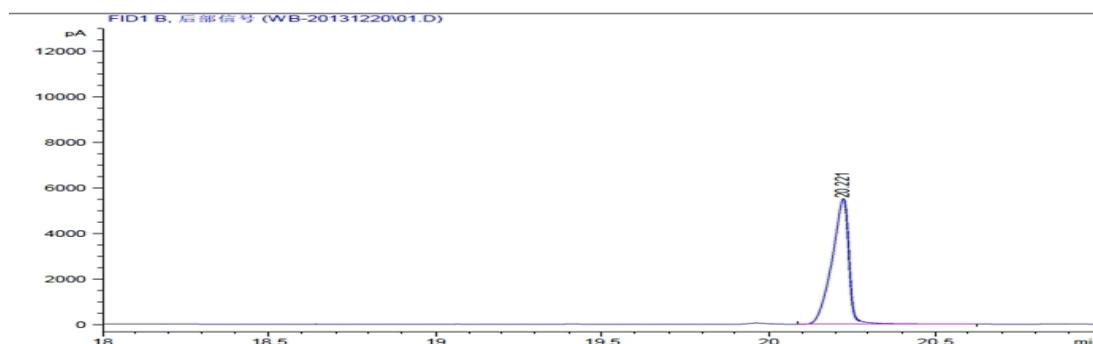


Figure 4 **2** (2,6,6-Trimethyl-cyclohex-2-ene-1,4-dione) $t_R = 20.22$ min

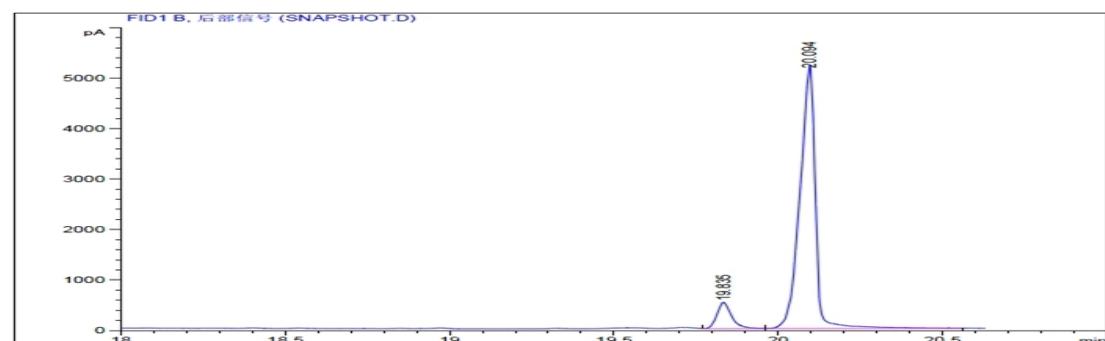


Figure 5 the reaction catalyzed by 0.1 mmol% $Rh_2(esp)_2$ under standard conditions

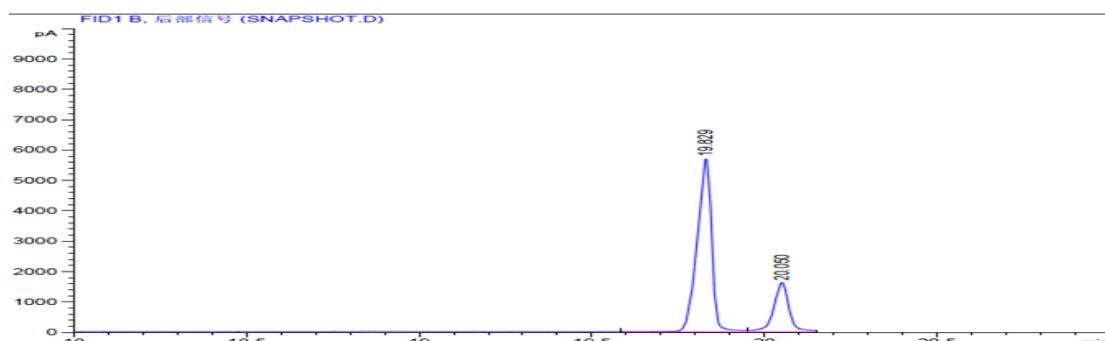


Figure 5 the reaction catalyzed by 0.1 mmol% $Rh_2(OAc)_4$ under standard conditions

The conversion of oxidation of α -1 (α -isophorone) catalyzed by 0.1 mol% Rh₂(esp)₂ and Rh₂(cap)₄

0.1 mol% catalyst	Conversion (%)		
	6 hr	10 hr	24 hr
Rh ₂ (esp) ₂	59	71	91
Rh ₂ (cap) ₄	38	43	49

The conversion of oxidation of α -1 (α -isophorone) catalyzed by 0.01 mol% Rh₂(esp)₂ and Rh₂(cap)₄

0.01 mol% catalyst	Conversion (%)		
	4 day	7 day	10 day
Rh ₂ (esp) ₂	62	78	88
Rh ₂ (cap) ₄	8	18	18

Rh₂(esp)₂ catalyst recycling experiment

Treatment of α -1 (3.6 g, 26 mmol) under standard conditions (0.1 mol % of Rh₂(esp)₂, 5.0 equiv T-HYDRO, nonesolvent) afforded **2** 96% conversion and 75% yield accompanied by the 10 mg Rh₂(esp)₂ (50% recovery rate) recovered through chromatographic purification. The recovered catalyst from the first reaction was then added to α -1 (1.8 g, 13 mmol), as well as another 5.0 equiv T-HYDRO, in order to initiate the second oxidation. When the recycling reaction finished, a 94% conversion, 77% yield of **2** resulted and the Rh₂(esp)₂ catalyst (6 mg, 60% recovery rate) was recovered. Further NMR study confirmed the recovered Rh₂(esp)₂ catalyst remained unchanged after two cycles.

Spectroscopic Data of the Catalysts and Isolated Products

Rh₂(esp)₂¹: ¹H NMR (400 MHz, CDCl₃) δ 7.09 (t, *J* = 7.5 Hz, 2H), 6.92 (s, 2H), 6.87 (dd, *J* = 7.5, 1.5 Hz, 4H), 2.66 (s, 8H), 1.03 (s, 24H). HRMS (ESI⁺) m/z calcd for C₃₂H₄₀O₈Rh₂ 759.0906 ([M+H]⁺) found 759.0913.

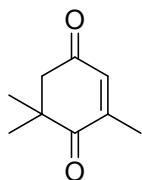
Rh₂(MPDP)₂² (acetone as the axial coordination): ¹H NMR (400 MHz, 1% v/v d₄-MeOH in CDCl₃) δ 7.09 (t, *J* = 7.5 Hz, 2H), 6.86 (dd, *J* = 7.6, 1.5 Hz, 4H), 6.80 (s, 2H), 2.77 – 2.67 (m, 8H), 2.36 – 2.28 (m, 8H). HRMS (ESI⁺) m/z calcd for C₂₄H₂₄O₈Rh₂ 646.9653 ([M+H]⁺) found 646.9633.

Rh₂(MPDP)(CO₂CF₃)₂³ (acetone as the axial coordination): ¹H NMR (400 MHz, 1% v/v d₄-MeOH in CDCl₃) δ 7.12 (t, J = 7.6 Hz, 1H), 6.89 (d, J = 7.5 Hz, 2H), 6.73 (s, 1H), 2.82 – 2.67 (m, 4H), 2.40 – 2.29 (m, 4H). HRMS (ESI⁺) m/z calcd for C₁₆H₁₂O₈F₆Rh₂ 652.8619 ([M+H]⁺) found 652.8630.

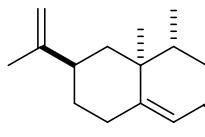
Rh₂(OAc)₄(IMes)⁴: ¹H NMR (400 MHz, CDCl₃) δ 7.23 (s, 2H), 6.80 (s, 4H), 2.23 (s, 12H), 2.22 (s, 6H), 1.55 (s, 12H). HRMS (ESI⁺) m/z calcd for C₂₉H₃₆N₂O₈Rh₂ 746.0582 ([M]⁺) found 746.0584.

Dirhodium(II) Complex I⁵ (acetone as the axial coordination): ¹H NMR (400 MHz, 3% v/v d₆-acetone in CDCl₃) δ 7.02 (t, J = 8.1 Hz, 2H), 6.46 (dd, J = 8.1, 2.2 Hz, 4H), 6.04 (s, 2H), 1.35 (s, 24H). HRMS (ESI⁺) m/z calcd for C₂₈H₃₂O₁₂Rh₂ 767.0076 ([M+H]⁺) found 766.8679.

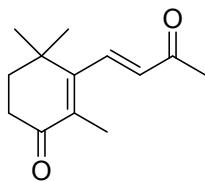
Dirhodium(II) Complex II⁵ (acetone as the axial coordination): ¹H NMR (400 MHz, 1% v/v d₄-MeOH in CDCl₃) δ 6.51 (s, 2H), 6.35 (s, 4H), 3.67 (s, 6H), 2.66 (m, 8H), 2.32 (m, 8H). HRMS (ESI⁺) m/z calcd for C₂₆H₂₈O₁₀Rh₂ 705.9787 ([M]⁺) found 705.9802.



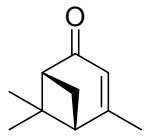
2,6,6-Trimethyl-cyclohex-2-ene-1,4-dione (2)⁶: ¹H NMR (400 MHz, CDCl₃) δ 6.54 (s, 1H), 2.70 (s, 2H), 1.99 (s, 3H), 1.22 (s, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 203.39, 197.68, 148.90, 137.02, 51.81, 45.13, 26.06, 16.78.



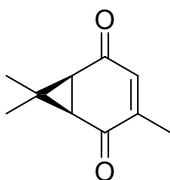
(+)-Nootkatone (4)⁷: ¹H NMR (400 MHz, CDCl₃) δ 5.77 (s, 1H), 4.74 (m, 1H), 4.72 (m, 1H), 1.74 (s, 3H), 1.11 (s, 3H), 0.96 (d, J = 6.8 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 199.66, 170.54, 149.08, 124.68, 109.23, 43.92, 42.07, 40.39, 39.33, 33.03, 31.62, 20.81, 16.85, 14.90.



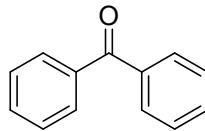
4-Oxo- β -ionone (6)⁸: ^1H NMR (400 MHz, CDCl_3) δ 7.20 (d, $J = 16.5$ Hz, 1H), 6.19 (d, $J = 16.4$ Hz, 1H), 2.52 (t, $J = 6.6$ Hz, 2H), 2.34 (s, 3H), 1.88 (t, $J = 7.0$ Hz, 2H), 1.79 (s, 3H), 1.18 (s, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 198.62, 197.46, 157.78, 140.36, 133.54, 131.41, 37.30, 35.55, 34.18, 27.97, 27.31, 13.43.



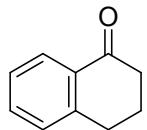
(S)-(-)- α -Verbinone (8)⁹: ^1H NMR (400 MHz, CDCl_3) δ 5.73 (m, 1H), 2.79 (m, 1H), 2.64 (dt, $J = 6.0, 1.7$ Hz, 1H), 2.41 (t, $J = 6.5$ Hz, 1H), 2.07 (d, $J = 9.1$ Hz, 1H), 2.01 (d, $J = 1.5$ Hz, 3H), 1.49 (s, 3H), 1.01 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 204.15, 170.43, 121.18, 57.60, 54.20, 49.73, 40.87, 26.59, 23.57, 22.04.



3,7,7-Trimethylbicyclo[4.1.0]hept-3-ene-2,5-dione (10)¹⁰: ^1H NMR (400 MHz, CDCl_3) δ 6.49 (m, 1H), 2.32 (m, 2H), 1.96 (d, $J = 4.0$ Hz, 3H), 1.32 (s, 3H), 1.31 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 194.97, 194.36, 149.97, 137.64, 39.82, 38.98, 33.55, 29.07, 16.20, 15.43.

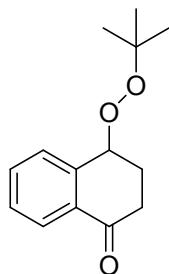


Benzophenone (12)¹¹: ^1H NMR (400 MHz, CDCl_3) δ 7.84 – 7.77 (m, 4H), 7.57-7.61 (m, 2H), 7.49 (t, $J = 7.5$ Hz, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 196.76, 137.60, 132.42, 130.06, 128.28.

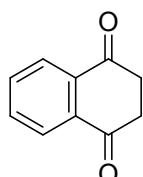


1-Tetralone (14)¹²: ^1H NMR (400 MHz, CDCl_3) δ 8.03 (d, $J = 7.8$ Hz, 1H), 7.46 (t, $J = 7.5$ Hz, 1H), 7.29 (t, $J = 7.8$ Hz, 1H), 7.25 (d, $J = 7.6$ Hz, 1H), 2.96 (t, $J = 6.1$ Hz, 2H), 2.65 (m, 2H), 2.13

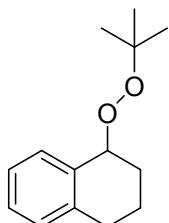
(m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 198.40, 144.50, 133.40, 132.59, 128.78, 127.14, 126.62, 39.17, 29.70, 23.29.



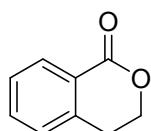
4-(tert-Butylperoxy)-3,4-dihydroronaphthalen-1(2H)-one (15)¹³: ^1H NMR (400 MHz, CDCl_3) δ 8.04 (d, $J = 7.9$ Hz, 1H), 7.53-7.59 (m, 2H), 7.45 (t, $J = 7.3$ Hz, 1H), 5.13 (t, $J = 4.0$ Hz, 1H), 2.93-3.01 (m, 1H), 2.52-2.62 (m, 2H), 2.28-2.36 (m, 1H), 1.24 (s, 9H). ^{13}C NMR (100 MHz, CDCl_3) δ 197.74, 140.01, 133.49, 132.35, 129.74, 129.09, 127.01, 80.57, 78.16, 33.96, 26.73, 26.45.



2,3-Dihydroronaphthalene-1,4-dione (16)¹⁴: ^1H NMR (300 MHz, CDCl_3) δ 8.04-8.07 (m, 2H), 7.73-7.76 (m, 2H), 3.09 (s, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 195.98, 138.67, 135.25, 134.28, 133.94, 126.73, 126.41, 37.54.

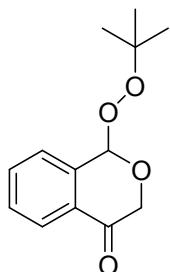


1-(tert-Butylperoxy)tetralin (17)¹⁵: ^1H NMR (400 MHz, CDCl_3) δ 7.45 (d, $J = 7.3$ Hz, 1H), 7.16-7.24 (m, 2H), 7.11 (d, $J = 7.3$ Hz, 1H), 5.00 (t, $J = 3.6$ Hz, 1H), 2.82 (dt, $J = 16.6, 4.8$ Hz, 1H), 2.76 – 2.64 (m, 1H), 2.35-2.41 (m, 1H), 1.92-2.03 (m, 1H), 1.71-1.84 (m, 2H), 1.30 (s, 9H). ^{13}C NMR (100 MHz, CDCl_3) δ 138.82, 133.41, 130.98, 129.03, 128.15, 125.61, 80.04, 78.79, 29.36, 27.08, 26.66, 18.10.

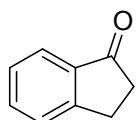


3,4-Dihydroisochromen-1-one (19)¹⁶: ^1H NMR (300 MHz, CDCl_3) δ 8.08 (d, $J = 7.7$ Hz, 1H), 7.53 (m, 1H), 7.38 (t, $J = 7.8$ Hz, 1H), 7.27 (d, $J = 7.3$ Hz, 1H), 4.52 (t, $J = 6.0$ Hz, 2H), 3.05 (t, J

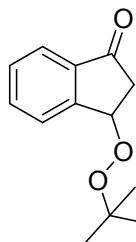
$= 6.0$ Hz, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 165.13, 139.55, 133.67, 130.40, 127.69, 127.24, 125.32, 67.31, 27.84.



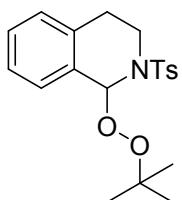
1-(tert-Butylperoxy)-1*H*-isochromen-4(3*H*)-one (20)¹⁷: ^1H NMR (300 MHz, CDCl_3) δ 8.02 (d, $J = 6.5$ Hz, 1H), 7.65 (dt, $J = 7.5$ Hz, 1.5 Hz, 1H), 7.55 (dt, $J = 7.0$ Hz, 1.4 Hz, 1H), 7.42 (d, $J = 7.1$ Hz, 1H), 6.20 (s, 1H), 4.85 (d, $J = 17.4$ Hz, 1H), 4.37 (d, $J = 17.4$ Hz, 1H), 1.33 (s, 9H). ^{13}C NMR (100 MHz, CDCl_3) δ 193.57, 135.97, 134.42, 130.14, 129.58, 127.14, 126.01, 99.44, 81.65, 66.26, 26.45.



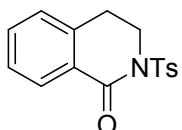
1-Indanone (22)¹⁸: ^1H NMR (300 MHz, CDCl_3) δ 7.76 (d, $J = 7.7$ Hz, 1H), 7.58 (m, 1H), 7.47 (d, $J = 7.7$ Hz, 1H), 7.36 (t, $J = 7.4$ Hz, 1H), 3.14 (t, $J = 5.6$ Hz, 2H), 2.69 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 207.09, 155.17, 137.09, 134.60, 127.28, 126.70, 123.72, 36.23, 25.81.



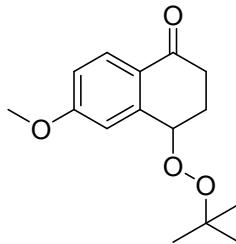
1-(t-Butylperoxy)indan-3-one (23)¹⁹: ^1H NMR (300 MHz, CDCl_3) δ 7.78 (m, 2H), 7.66 (t, $J = 7.5$ Hz, 1H), 7.51 (t, $J = 7.4$ Hz, 1H), 5.65 (dd, $J = 6.4, 2.4$ Hz, 1H), 2.99 (dd, $J = 19.2, 6.4$ Hz, 1H), 2.82 (dd, $J = 19.1, 2.5$ Hz, 1H), 1.27 (s, 9H). ^{13}C NMR (100 MHz, CDCl_3) δ 207.09, 155.17, 137.09, 134.60, 127.28, 126.70, 123.72, 36.23, 25.81.



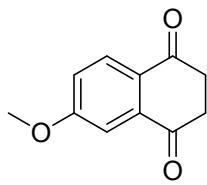
1-(tert-Butylperoxy)-2-tosyl-1,2,3,4-tetrahydroisoquinoline (25)²⁰: ¹H NMR (400 MHz, CDCl₃) δ 7.84 (d, *J* = 8.3 Hz, 2H), 7.40 (d, *J* = 8.8 Hz, 1H), 7.22-7.29 (m, 4H), 7.11 (d, *J* = 7.2 Hz, 1H), 6.55 (s, 1H), 3.80 (dd, *J* = 14.0, 4.9 Hz, 1H), 3.51-3.58(m, 1H), 2.90-2.99 (m, 1H), 2.68 (d, *J* = 20.2 Hz, 1H), 2.40 (s, 3H), 1.19 (s, 9H). ¹³C NMR (101 MHz, CDCl₃) δ 143.04, 138.13, 135.35, 130.16, 129.40, 129.27, 129.08, 127.88, 127.66, 126.32, 85.45, 80.88, 38.58, 28.16, 26.48, 21.49.



2-Tosyl-3,4-dihydroisoquinolin-1(2H)-one (26)²¹: ¹H NMR (400 MHz, CDCl₃) δ 7.99 (d, *J* = 8.0 Hz, 1H), 7.98 (d, *J* = 8.4 Hz, 2H), 7.47 (t, *J* = 5.8 Hz, 1H), 7.33 (d, *J* = 8.6 Hz, 3H), 7.21 (d, *J* = 7.6 Hz, 1H), 4.27 – 4.20 (m, 2H), 3.13 (t, *J* = 6.2 Hz, 2H), 2.42 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 163.50, 144.76, 139.25, 136.15, 133.50, 129.43, 129.21, 128.59, 128.25, 127.41, 44.74, 28.97, 21.68.



4-(tert-Butylperoxy)-6-methoxy-3,4-dihydronaphthalen-1(2H)-one (28): ¹H NMR (300 MHz, CDCl₃) δ 7.51 (d, *J* = 2.8 Hz, 1H), 7.44 (d, *J* = 8.5 Hz, 1H), 7.12 (dd, *J* = 8.4, 2.8 Hz, 1H), 5.08 (t, *J* = 3.6 Hz, 1H), 3.85 (s, 3H), 2.97 (m, 1H), 2.58 (m, 2H), 2.25 (m, 1H), 1.24 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 197.80, 160.21, 133.65, 132.43, 131.49, 121.36, 109.24, 80.45, 55.55, 33.64, 26.47. HRMS (ESI⁺) m/z calcd for C₁₅H₁₂O₄ 265.1434 ([M+H]⁺) found 265.1437.



6-Methoxy-2,3-dihydronaphthalene-1,4-dione (29)²²: ¹H NMR (400 MHz, CDCl₃) δ 8.01 (d, *J* = 8.7 Hz, 1H), 7.44 (d, *J* = 2.7 Hz, 1H), 7.22 (dd, *J* = 8.7, 2.7 Hz, 1H), 3.93 (s, 3H), 3.01-3.09 (m, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 196.27, 194.80, 164.30, 137.34, 129.23, 128.92, 121.67, 108.87, 55.93, 37.75, 37.16.

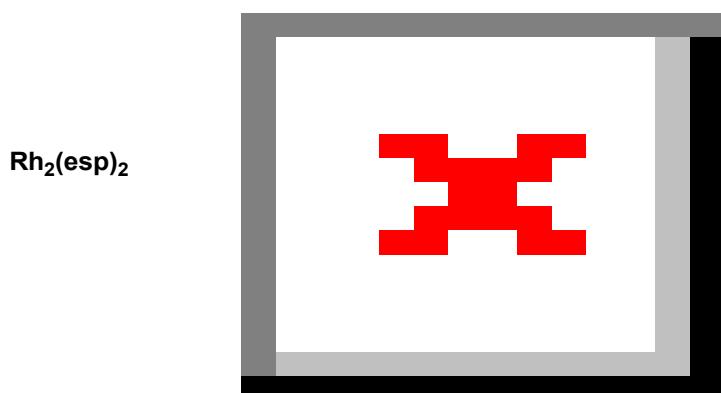
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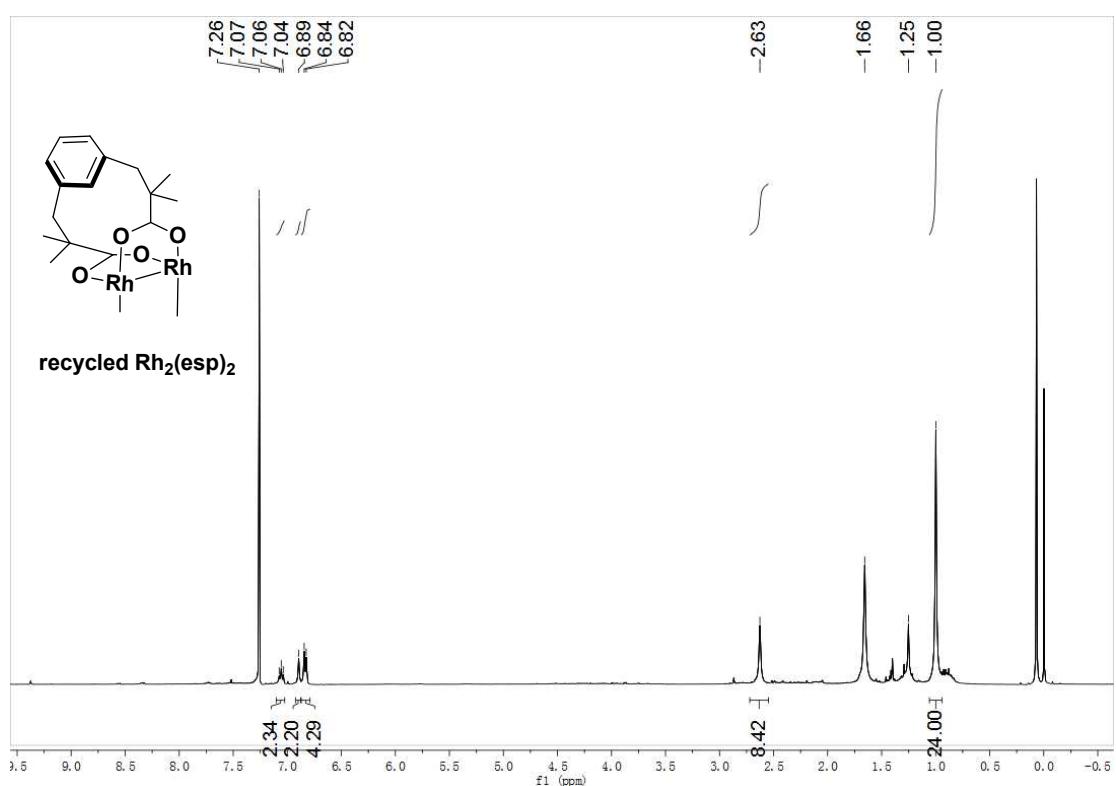
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¹H and ¹³C NMR Spectrum of compounds

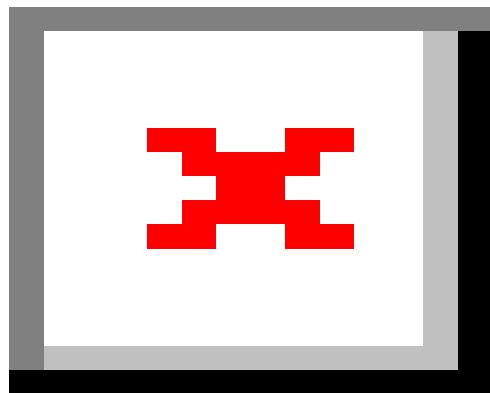
¹H NMR of Rh₂(esp)₂



¹H NMR of recycled Rh₂(esp)₂



$\text{Rh}_2(\text{MPDP})_2$



$\text{Rh}_2(\text{MPDP})(\text{CO}_2\text{CF}_3)_2$

