SUPPLEMENTARY INFORMATION

Rhenium-Containing Compound(PyHReO4): Synthesis, Characterization and Catalytic Application in Olefin Epoxidation and Baeyer-Villiger Oxidation

HAN WANG*, YAN ZHAO, DONG ZHANG, YUCAI LI, YINGNAN DONG, SHUAIJIE WANG, SHIWEI SONG, JIAN WANG and GANG WANG
School of Renewable Energy, Shenyang Institute of Engineering, Shenyang110136, Liaoning, China

E-mail: wanghan_1205@sina.com

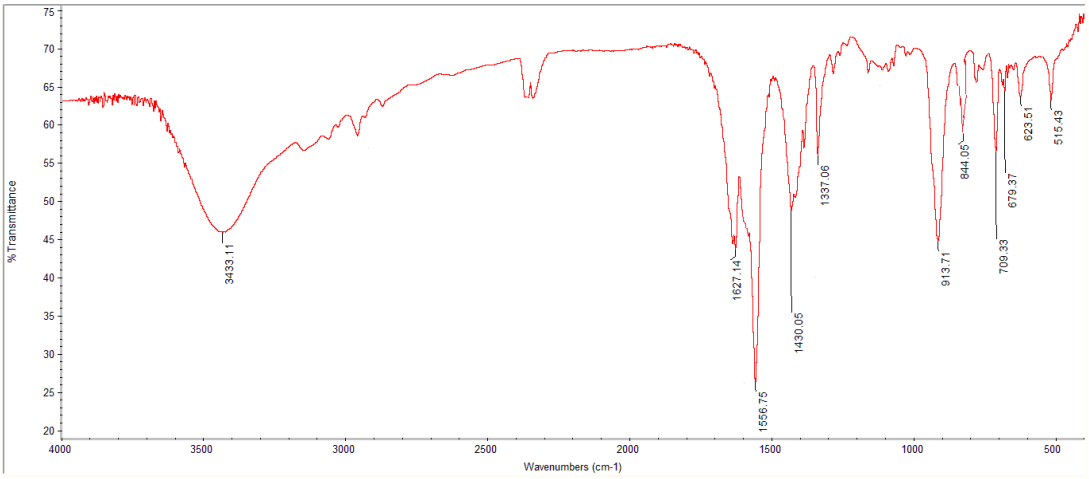


Fig. 1 FT-IR spectrum of $PyHReO_4$

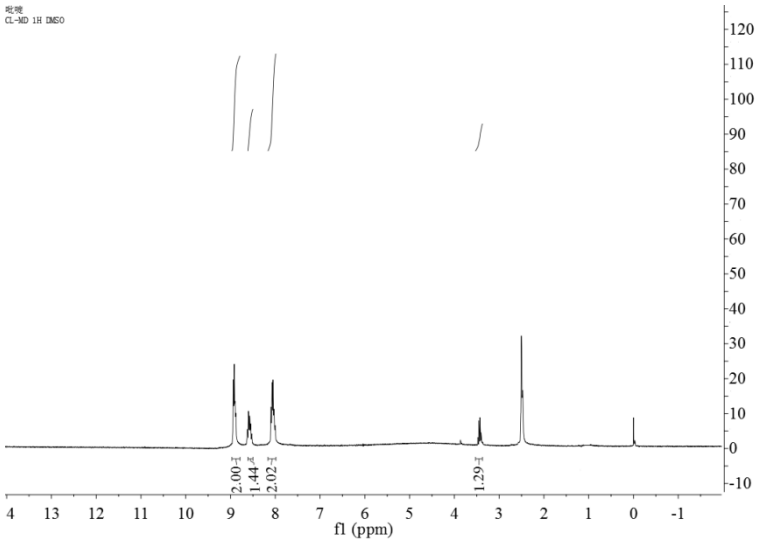
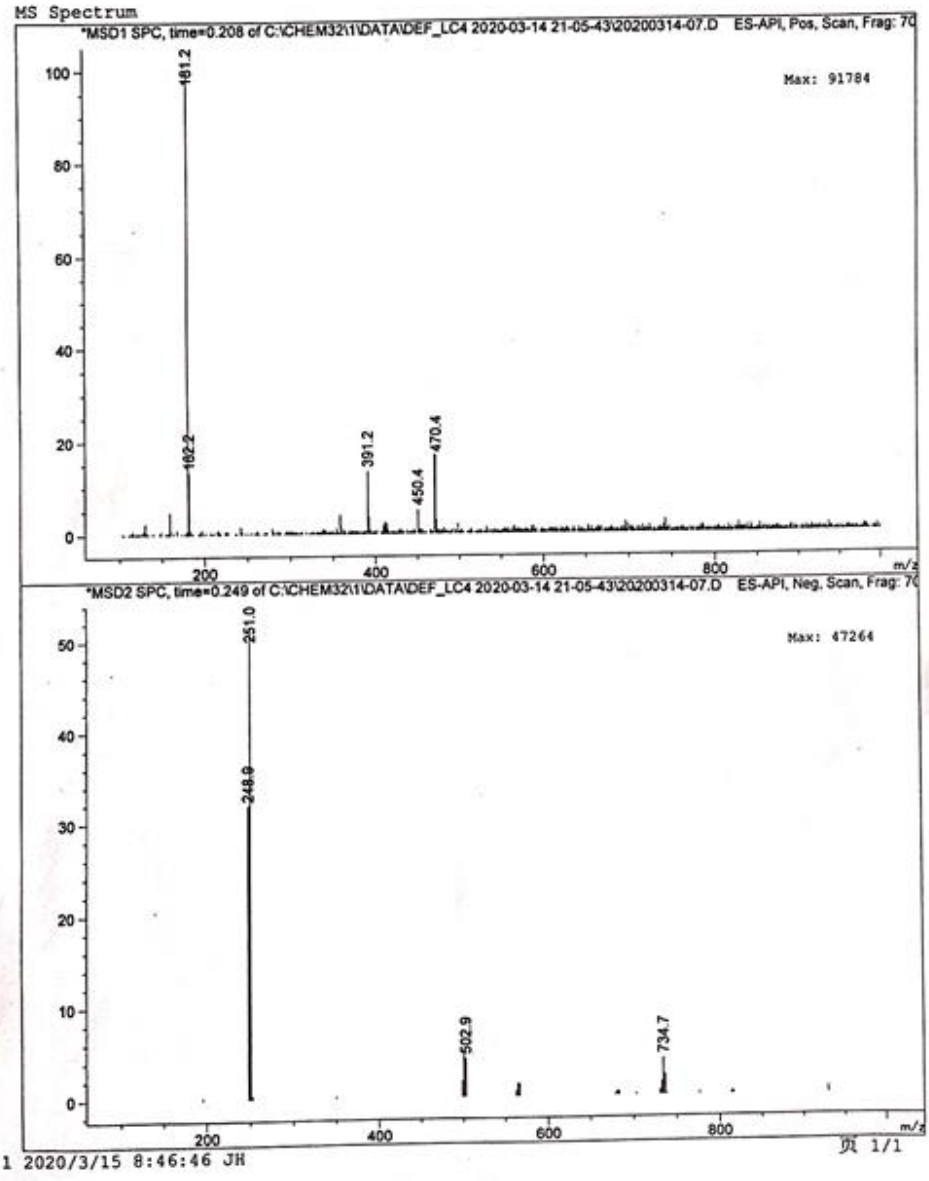


Fig. 2 ^{1}HNMR(300 MHz,DMSO) spectrum of $PyHReO_4$

操作者 : JH
仪器 : 仪器 1
进样日期 : 2020/3/14 21:39:43
序列行 : 7
位置 : 样品瓶 37
进样次数 : 1
进样量 : 5.000 µl

采集方法 : C:\CHEM32\1\DATA\DEF_LC4 2020-03-14 21-05-43\ESI-B-LAMPOFF-2MIN.M
最后修改 : 2020/3/12 11:11:53 : JH
分析方法 : D:\METHODS\ESI-B-LAMPOFF-2MIN.M
最后修改 : 2020/3/12 11:11:53 : JH
方法信息 : ESI-B-LAMPOFF-2MIN



仪器 1 2020/3/15 8:46:46 JH 页 1/1

Fig. 3 ESI-MS(Acetonitrile) spectra of $PyHReO_4$

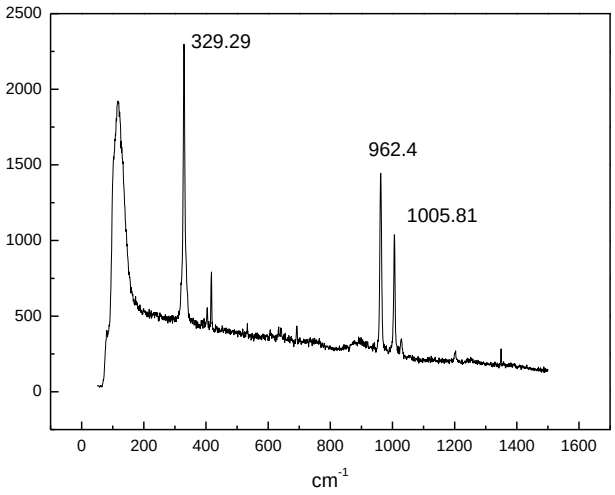


Fig. 4 The Raman spectra of $PyHReO_4$

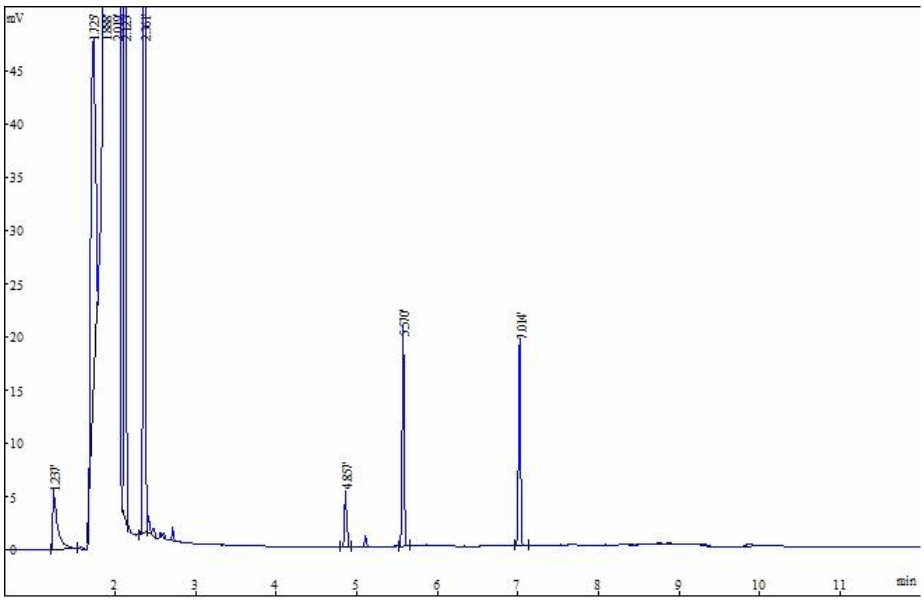


Fig. 5 GC data of optimal experiment on cyclooctene epoxidation

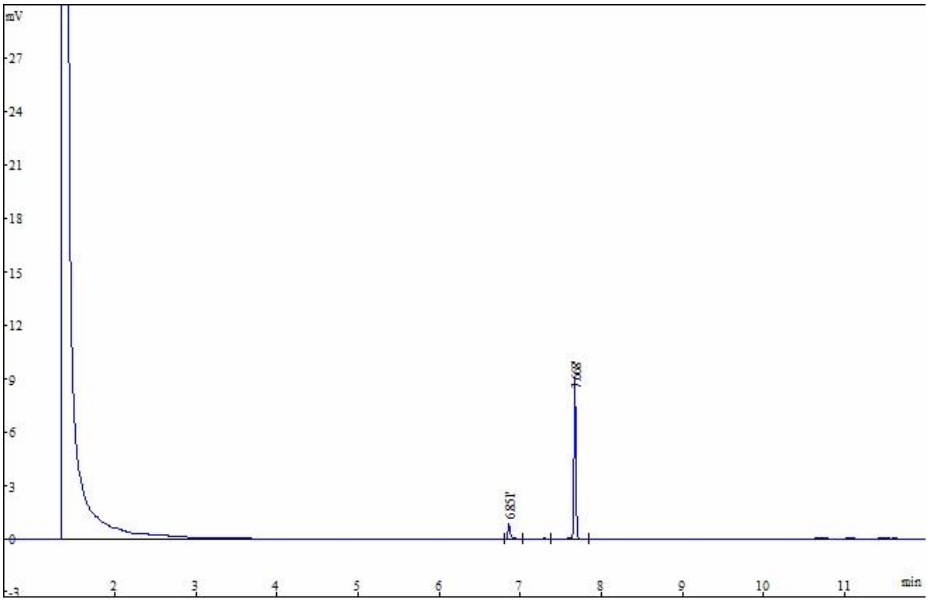


Fig. 6 GC data of optimal experiment of 2-adamantanone on Baeyer-Villiger oxidation

Fig. 7 Crystal photo of $PyHReO_4$

Abstract

Computing details

Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997).

References

NOT FOUND

(shelxl)

Crystal data

$C_5H_6NO_4Re$	V = 1491.7 (3) Å^3
M_r = 330.31	Z = 8
?, ?	F(000) = 1200
a = 7.230 (1) Å	D_x = 2.941 Mg m^{-3}
b = 12.1070 (15) Å	Mo $K\alpha$ radiation, λ = 0.71075 Å
c = 17.0420 (18) Å	μ = 16.26 mm^{-1}
α = 90°	T = 113 K
β = 90°	0.24 × 0.22 × 0.18 mm
γ = 90°	

Data collection

Radiation source: fine-focus sealed tube	R_{int} = 0.125
graphite	θ_{max} = 30.1°, θ_{min} = 2.4°
18604 measured reflections	h = −10→10
2178 independent reflections	k = −17→16
2160 reflections with $I > 2\sigma(I)$	l = −23→23

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.046	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.121	$w = 1/[\sigma^2(F_o^2) + (0.0637P)^2 + 19.3911P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.13	$(\Delta/\sigma)_{max}$ = 0.001
2178 reflections	$\Delta\rho_{max}$ = 3.81 e Å^{-3}
101 parameters	$\Delta\rho_{min}$ = −6.16 e Å^{-3}

0 restraints	Extinction correction: *SHELXL*, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.00086 (15)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Re1	0.11765 (3)	0.26308 (2)	0.363573 (14)	0.00525 (15)
O1	0.1589 (8)	0.4036 (5)	0.3585 (3)	0.0162 (10)
O2	0.0227 (8)	0.2185 (4)	0.2752 (3)	0.0162 (10)
O3	0.3252 (8)	0.1937 (5)	0.3810 (3)	0.0216 (11)
O4	−0.0328 (8)	0.2374 (4)	0.4392 (3)	0.0152 (10)
N1	0.6583 (8)	0.0831 (5)	0.3518 (3)	0.0099 (10)
H1	0.5895	0.1357	0.3313	0.012*
C1	0.7629 (8)	0.0209 (5)	0.3037 (4)	0.0096 (11)
H1A	0.7614	0.0339	0.2488	0.012*
C2	0.8732 (9)	−0.0626 (7)	0.3346 (4)	0.0133 (14)
H2	0.9475	−0.1070	0.3011	0.016*
C3	0.8729 (8)	−0.0801 (7)	0.4157 (4)	0.0128 (13)
H3	0.9482	−0.1362	0.4379	0.015*
C4	0.7614 (8)	−0.0146 (5)	0.4641 (4)	0.0108 (12)
H4	0.7587	−0.0264	0.5192	0.013*
C5	0.6549 (9)	0.0678 (6)	0.4300 (4)	0.0114 (11)
H5	0.5794	0.1136	0.4620	0.014*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Re1	0.00068 (18)	0.0073 (2)	0.0078 (2)	0.00115 (7)	0.00023 (6)	−0.00003 (7)
O1	0.017 (2)	0.009 (2)	0.022 (2)	−0.004 (2)	0.0004 (18)	0.0004 (17)
O2	0.019 (2)	0.019 (2)	0.011 (2)	−0.004 (2)	−0.0001 (18)	−0.0027 (18)
O3	0.015 (3)	0.029 (3)	0.021 (2)	0.014 (2)	0.000 (2)	0.003 (2)
O4	0.018 (3)	0.015 (2)	0.013 (2)	−0.0031 (18)	0.003 (2)	0.0023 (16)
N1	0.003 (2)	0.012 (3)	0.014 (2)	−0.002 (2)	0.0013 (19)	0.002 (2)
C1	0.006 (2)	0.011 (3)	0.012 (3)	0.000 (2)	0.0007 (18)	−0.002 (2)
C2	0.008 (3)	0.013 (3)	0.019 (3)	0.001 (2)	0.002 (2)	−0.006 (3)
C3	0.010 (3)	0.012 (3)	0.017 (3)	0.000 (2)	0.000 (2)	0.002 (3)

C4	0.005 (2)	0.016 (3)	0.012 (3)	0.002 (2)	0.0007 (18)	−0.001 (2)
C5	0.003 (2)	0.017 (3)	0.015 (3)	−0.003 (2)	0.000 (2)	−0.001 (2)

Geometric parameters (Å, º)

Re1—O4	1.715 (5)	C1—H1A	0.9500
Re1—O1	1.730 (6)	C2—C3	1.398 (10)
Re1—O2	1.740 (5)	C2—H2	0.9500
Re1—O3	1.745 (5)	C3—C4	1.399 (9)
N1—C1	1.345 (8)	C3—H3	0.9500
N1—C5	1.347 (9)	C4—C5	1.387 (9)
N1—H1	0.8800	C4—H4	0.9500
C1—C2	1.391 (9)	C5—H5	0.9500
O4—Re1—O1	109.0 (2)	C1—C2—C3	118.9 (6)
O4—Re1—O2	110.1 (3)	C1—C2—H2	120.6
O1—Re1—O2	109.2 (2)	C3—C2—H2	120.6
O4—Re1—O3	109.3 (3)	C2—C3—C4	119.9 (7)
O1—Re1—O3	109.5 (3)	C2—C3—H3	120.1
O2—Re1—O3	109.7 (3)	C4—C3—H3	120.1
C1—N1—C5	122.4 (6)	C5—C4—C3	118.7 (7)
C1—N1—H1	118.8	C5—C4—H4	120.6
C5—N1—H1	118.8	C3—C4—H4	120.6
N1—C1—C2	120.0 (6)	N1—C5—C4	120.2 (6)
N1—C1—H1A	120.0	N1—C5—H5	119.9
C2—C1—H1A	120.0	C4—C5—H5	119.9
C5—N1—C1—C2	−0.2 (10)	C2—C3—C4—C5	−0.9 (10)
N1—C1—C2—C3	0.0 (10)	C1—N1—C5—C4	−0.2 (10)
C1—C2—C3—C4	0.6 (10)	C3—C4—C5—N1	0.7 (9)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3	0.88	2.20	2.800 (8)	125
C5—H5···O4^{i}	0.95	2.60	3.517 (9)	163
N1—H1···O2^{ii}	0.88	2.13	2.887 (8)	144
C5—H5···O4^{i}	0.95	2.60	3.517 (9)	163

Symmetry codes: (i) *x*+1/2, −*y*+1/2, −*z*+1; (ii) *x*+1/2, *y*, −*z*+1/2.