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Methylrhenium trioxide as a catalyst for oxidations with molecular oxygen and for oxygen transfer

Zuolin Zhu, James H. Espenson *

Ames Laboratory and Department of Chemistry, Iowa State University, Ames, IA 50011, USA

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Abstract

Methylrhenium trioxide (MTO) was found to be a good catalyst for the oxidation of tertiary phosphines by molecular oxygen at room temperature. Evidence is given that an intermediate Re(V) compound – CH_3ReO_2, or the adduct $CH_3ReO_2 \cdot O{=}PPh_3$ – is involved. The deoxygenation of epoxides, sulfoxides, N-oxides, triphenylarsine oxide and triphenylstibine oxide at room temperature was also catalyzed by MTO, with triphenylphosphine as the oxygen acceptor. A plausible reaction mechanism involves phosphine attack at a compound formed between MTO and the epoxide or other oxygen-donor compound.

Keywords: Catalysis; Phosphines; Oxidation; Rhenium; Oxygen

1. Introduction

Transition metal–oxo complexes are of great relevance to many catalytic oxidation processes and to oxygen atom transfer between substrates [1]. Methylrhenium trioxide (CH_3ReO_3, abbreviated as MTO), is a stable compound prepared from dirhenium heptoxide and tetramethyltin [2]. It acts as an efficient homogeneous oxidation catalyst for hydrogen peroxide in both aqueous and organic solvents. With hydrogen peroxide as the oxidant, MTO catalyzes olefin epoxidations [3,4], conversion of thiolatocobalt to sulfenatocobalt [5], oxidations of organic sulfides [6], phosphines, triphenylarsine, and triphenylstibine [7], and tertiary amines to the corresponding oxides [8], and for the conversion of aniline to nitrosobenzene [8]. With **S** representing such a substrate, all of these reactions can be abbreviated as:

$$S + H_2O_2 \xrightarrow{\text{cat. MTO}} S{=}O + H_2O \qquad (1)$$

Since both the activation of molecular oxygen and oxygen transfer are important industrially and biologically, the catalytic properties of many transition metal oxo complexes have been studied, including ruthenium(V) [9], molybdenum(V) [10], ruthenium(IV) [11], and rhenium(V) [12]. These oxo complexes and the hexanuclear carbonyl cluster $Rh_6(CO)_{16}$ [13] are capable of activating molecular oxygen or transferring an oxygen atom. This type of catalytic process for methylrhenium trioxide remains unexplored.

We have found that MTO catalyzes the oxidation of tertiary phosphines by molecular oxygen. Also, we have examined the transfer of the oxygen atom from epoxides, sulfoxides, aromatic tertiary

* Corresponding author.

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amine oxides, triphenylarsine oxide, and triphenylstibine oxide. In each case the oxygen acceptor was triphenylphosphine.

2. Results

2.1. Catalyzed phosphine oxidation with molecular oxygen

The oxidation of tertiary phosphines was conducted in benzene at room temperature. Two different methods were used. First, the solutions containing MTO and the triarylphosphines were opened to air with stirring. One day later, substantial yields of phosphine oxides were recorded. The yields differ with the substrate, as given in Table 1. After two days, however, all of the phosphines had been converted to their oxides in yields of >95%. Second, pure oxygen was used instead of air. In that case the reactions reached completion within six hours or less, with yields of >95%. An independent reaction was carried out in which MTO was not used: triphenyl phosphine was dissolved in benzene and exposed to air and stirred. After one week, triphenyl phosphine oxide was not detected from this reaction.

The reactions under pure oxygen at room temperature were monitored by ^{1}H-NMR. During the reaction the signal of the phosphine decreased as that of the oxide appeared. Without oxygen, only one signal from the rhenium present was seen at $\delta = 1.212$ ppm. This corresponds to rhenium(V), perhaps $[CH_3ReO_2 \cdot OPPh_3]$ or simply $[CH_3ReO_2]$ [14], which will be abbreviated $\mathbf{V_A}$. A new peak appeared at $\delta = 1.237$ ppm after the solution was flushed with oxygen. Isolation of the latter species is in progress.

Table 1
Product yields from the oxidation of triarylphosphines with air, catalyzed by MTO [a]

$(4\text{-R-}C_6H_4)_3P$ R=	% Yield 24 h	48 h
H	62	>97
CH_3	71	100
Cl	53	>96
CH_3O	80	100

[a] In benzene solution at room temperature, with phosphine: MTO ≈ 10:1.

2.2. Oxygen transfer reactions

The reactions described in this section were catalyzed by MTO. This was confirmed by carrying out controls without MTO; in each case, no oxygen transfer reaction was observed even after one week.

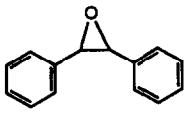
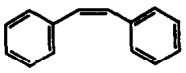
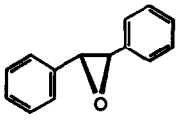
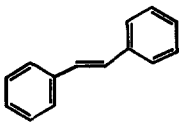
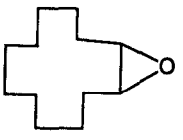
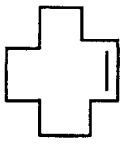
Treatment of an epoxide with triphenylphosphine in the presence of MTO under Ar at room temperature results in the deoxygenation of the epoxide and the formation of an olefin in high yield. The reaction preserves the relative stereochemistry about the C–C bond of the epoxide. The results are given in Table 2, which lists the epoxide taken, the olefin obtained, and its yield.

Oxygen abstraction from epoxides was also monitored by ^{1}H-NMR. Upon mixing the epoxide and MTO in benzene at room temperature, the signal corresponding to a dialkoxylrhenium complex (or rhenium glycolate) was observed [16]. Upon addition of triphenylphosphine, signals corresponding to Ph_3PO, olefin, and MTO were seen. Crystals were also isolated from the reaction mixture. They had the same ^{1}H-NMR spectrum as the dialkoxylrhenium complex in solution [16].

Treated analogously in benzene, several sulfoxides reacted with triphenylphosphine at room temperature to give the sulfide as the only product. The reaction was studied by ^{1}H-NMR at room temperature. A 1:1 solution of the sulfoxide and MTO showed that the CH_3 group of rhenium after one hour had shifted downfield about 0.1 ppm. Upon addition of triphenylphosphine, the signals corresponding to triphenylphosphine oxide and sulfide appeared immediately (see Table 3).

MTO also catalyzes oxygen transfer from tertiary amine oxides to triphenylphosphine, forming the amine and triphenylphosphine oxide. This reaction also occurs at room temperature under argon. The results are shown in Table 4. As for the epoxides, this reaction was investigated by ^{1}H-

Table 2
Deoxygenation of epoxides with triphenylphosphine, catalyzed by MTO [a]

Substrate	Olefin product	Yield (%)
styrene epoxide	styrene	83
cyclohexene epoxide	cyclohexene	71 [b]
		87
		85
		79
		81 [b]

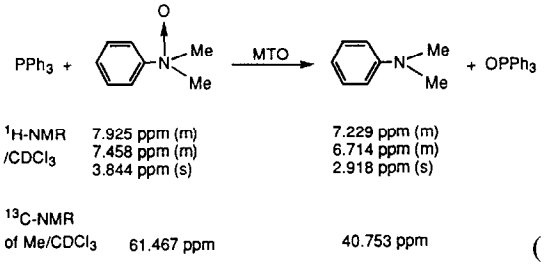
[a] In benzene at room temperature with epoxide: MTO ≈ 10:1.
[b] Product yields were calculated from ^{1}H-NMR.

Table 3
Deoxygenation of sulfoxides with triphenylphosphine, catalyzed by MTO [a]

Substrate	Sulfide produced	Yield (%)
PhS(O)CH=CH$_2$	PhSCH=CH$_2$	73
Ph$_2$SO	Ph$_2$S	66
PhS(O)Me	PhSMe	71
(Pr^i)$_2$SO	(Pr^i)$_2$S	75
(Bu^n)$_2$SO	(Bu^n)$_2$S	77
(*p*-CH$_3$C$_6$H$_4$)$_2$SO	(*p*-CH$_3$C$_6$H$_4$)$_2$S	65
(*p*-ClC$_6$H$_4$)$_2$SO	(*p*-ClC$_6$H$_4$)$_2$S	59

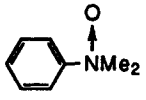
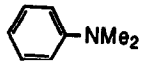
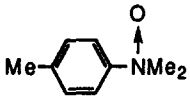
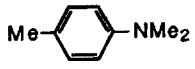
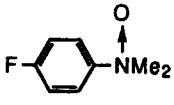
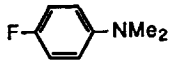
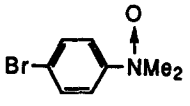
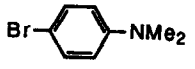
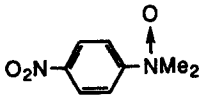
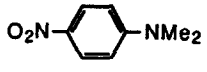
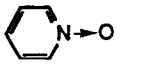
[a] In benzene at room temperature, with sulfoxide: MTO ≈ 10:1.

NMR, ^{13}C-NMR and GC–MS for *N*,*N*-dimethyl aniline *N*-oxide; see Eq. 2. GC–MS was employed in monitoring the reactions and for product identification for the other amine oxides. A mixture of MTO and the amine oxide in benzene at room temperature also shows a chemical shift about 0.1 ppm downfield of the methyl group of MTO which we believe is due to the effect of the substrates used. After the reaction has finished, MTO is still active. This was confirmed by the continuation of the catalytic process when more reactants were added.



(2)

Table 4
Deoxygenation of tertiary amine oxides with triphenylphosphine, catalyzed by MTO [a]

Substrate	Amine product	Yield (%)
		78
		81
		74
		70
		65
		77

[a] In benzene at room temperature, with a mole ratio *N*-oxide: MTO ≈ 10:1.

Unlike the oxygen transfer reactions reported previously [15], the deoxygenations of triphenylarsine oxide and triphenylstibine oxide by Ph_3P are very fast when catalyzed by MTO. Both reactions were finished within 30 min. at room temperature under argon; triphenylarsine and triphenylstibine were formed almost quantitatively. The identities of these products were confirmed spectroscopically; see Eq. 3.

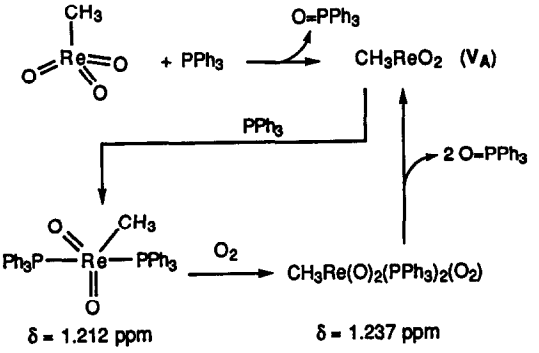
PPh_3	+	$OEPh_3$	$\xrightarrow{MTO}$	EPh_3	+	$OPPh_3$
1H-NMR		7.85 ppm(m)		7.35 ppm(m)		E = As
/$CDCl_3$		7.74 ppm(m)				(3)
/C_6D_6				7.42 ppm		E = Sb
				7.06 ppm		

In the absence of triphenylphosphine, the ^{1}H-NMR signal of the methyl group of MTO shifted from 1.196 ppm to 1.390 (E = As) or 1.410 (E = Sb) after addition of excess triphenylarsine oxide or triphenylstibine oxide in benzene. These species are stable at room temperature under argon, where there is no change within three days. In addition about 2% of the MTO decomposed to methanol; but the extent of decomposition did not increase with time in the absence of triphenylphosphine.

3. Discussion

3.1. The oxidation of phosphines with molecular oxygen

The absence of an NMR signal for the methyl group of MTO or other possible oxidants during the reactions was noted. The well-known mono-η^2-peroxide, $CH_3Re(O)_2(O_2)$ **A**, and the di-η^2-peroxide, $CH_3Re(O)(O_2)_2$ **B**, if present at all, remained undetected. This finding suggested a reaction pathway unlike that for catalytic oxidations that use hydrogen peroxide as the oxidant [5,7,8,14]. In contrast, the oxidations of phosphines with molecular oxygen do not seem to occur by way of **A** or **B** formed from hydrogen peroxide. (Later, however, this statement will be revised so as to admit the possibility that **A** might



Scheme 1.

be involved, but simply present at too low a concentration for detection.) From the ^{1}H-NMR data and results in the literature [17], we suggest that this reaction may follow a mechanism that involves an oxygen-containing intermediate, Scheme 1.

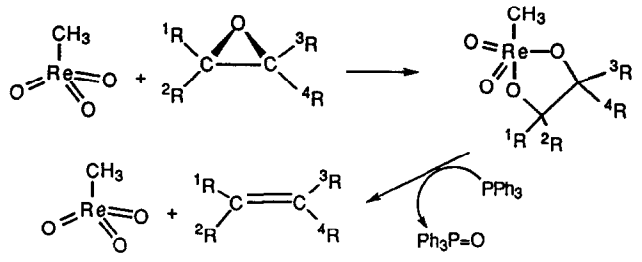
The incorporation into a substrate of an oxygen atom from molecular oxygen is important both industrially and biologically. Phosphines act as blood poisons, which may arise from the reaction with oxygen in the presence of some hemeproteins [18]. The in-depth study of this reaction may offer a ready explanation of the toxic effect.

3.2. Deoxygenation of epoxides

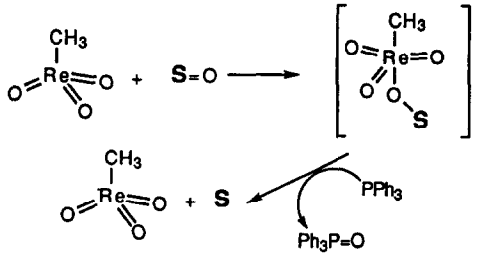
This reaction is important in both synthesis and structural determinations [19–22]. It provides a simple (one pot) method that proceeds in high yield under mild conditions. This reaction is believed to occur by a mechanism that features the participation of a dialkoxylrhenium complex (or rhenium glycolate), as given in Scheme 2.

3.3. Deoxygenation of sulfoxides and tertiary amine oxides

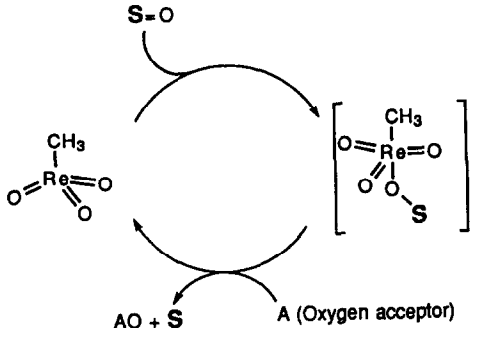
Oxygen atom transfer reactions have received renewed attention in the last few years because of their importance in biological systems [23]. A number of methods have so far been developed for this purpose [24,25]. Unlike the method given here, the methods previously reported need either a long reaction time [26] or a higher temperature



Scheme 2.



Scheme 3.



Scheme 4.

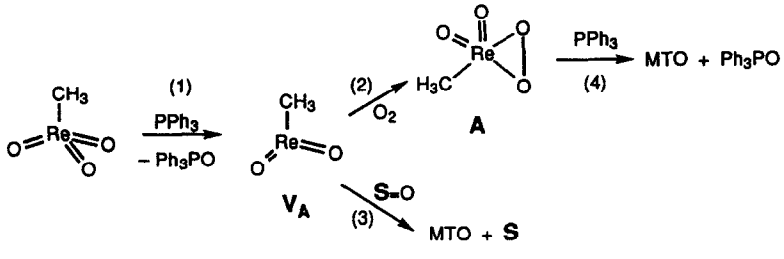
[27]. By analogy to the mechanism suggested for the deoxygenation of epoxides, the deoxygenation of sulfoxides and of *N*-oxides proceeds by a pathway in which the oxides first coordinate to MTO, and then (likely in > 1 step) it transfers an oxygen atom to triphenylphosphine. This set of reactions is depicted in Scheme 3.

3.4. Deoxygenation of triphenylarsine and triphenylstibine oxides

These reactions appear to follow a mechanism similar to that of the deoxygenation of sulfoxides and *N*-oxides. First, MTO coordinates $OAsPh_3$ or $OSbPh_3$, then PPh_3 reacts with this complex to form $AsPh_3$ or $SbPh_3$. The formation of methanol may arise from a very reactive oxidizing species in triphenylarsine oxide through an intermediate like '**A**', referred to previously.

These results point to MTO being an effective catalyst for several oxygen transfer reactions. The net stoichiometric scheme applicable to all of these reactions can be depicted as in Scheme 4.

We would also mention another set of reactions that may account for *all* of the processes reported herein. It entails a rhenium(V) intermediate $CH_3Re(O)_2$, which we shall abbreviate as $\mathbf{V_A}$ (a term arising from the role of this species in other reactions that will be reported independently). The actual formula of $\mathbf{V_A}$ might instead be $CH_3Re(O)_2 \cdot OPPh_3$, a species referred to above. The postulate is this: $\mathbf{V_A}$ forms first, in a reaction between MTO and PPh_3. It partitions between alternative reactions, governed by the presence of oxygen or of the substrate oxide. Included is a



Scheme 5.

reaction known from earlier work [7], between the η^2-peroxorhenium complex, **A**, and the phosphine, shown as step (4) in Scheme 5, which represents an alternative but reasonable picture for the general substrate S=O. At the present time, no clear resolution among these alternatives is at hand, although this and related chemistry remains an active endeavor.

These investigations have shown that MTO can act as an effective catalyst for a number of deoxygenation and oxygen transfer reactions, given a proper oxygen acceptor. Current studies are underway to extend the scope of this reaction.

4. Experimental section

4.1. Materials

The triarylphosphines, epoxides, sulfoxides, pyridine-*N*-oxide, and triphenylarsine oxide were purchased. The other *N*-oxides were obtained from our previous study [8]. 2,3-Dimethylbutene epoxide was prepared from 2,3-dimethyl-2-butene [28]. Triphenylstibine oxide was prepared by oxidizing triphenylstibine with hydrogen peroxide, catalyzed by MTO [7]. Methylrhenium trioxide was synthesized from dirhenium heptoxide and tetramethyltin in the presence of perfluoroglutaric anhydride [2]. Benzene was purified by a standard method [29].

4.2. Oxidation of phosphines

Method A

The tertiary phosphine (30 mmol) and MTO (3 mmol) were dissolved in 100 ml benzene, and the solution was stirred in air at room temperature. The reaction was monitored by TLC, ^{1}H-NMR, and ^{31}P-NMR; to do this several drops of the reaction solution were dried in a stream of argon and the residue dissolved in C_6D_6 for NMR. After 48 h the solvent was removed under vacuum and the residue recrystallized from methanol.

Method B

The materials in the amounts described above were dissolved in 100 ml benzene, then flushed with oxygen at room temperature. The reaction was monitored as previously described. After 6 h, the solvent was removed and the products identified by MS, ^{1}H-NMR, or ^{31}P-NMR [30–34].

Triphenylphosphine oxide. ^{1}H-NMR/$CDCl_3$: δ 7.68 ppm (m), 7.43 ppm (m). MS (EI): *m/e* 278, (CI, ammonia); 279 ($M+H^+$) and 296 ($M+NH_4^+$).

(p-Me–Ph)$_3$PO. ^{1}H-NMR/$CDCl_3$: δ 7.26 ppm (m), 7.533 ppm (m) and 2.38 ppm (s). MS (EI) *m/e* 320, (CI, ammonia), *m/e* 321 ($M+H^+$) and *m/e* 338 ($M+NH_4^+$).

(p-MeO–Ph)$_3$PO. ^{1}H-NMR/$CDCl_3$: δ 7.55 ppm (m), 6.97 ppm (m) and 3.84 ppm (s). MS (EI): *m/e* 368, (CI, ammonia), *m/e* 369 ($M+H^+$) and *m/e* 386 ($M+NH_4^+$).

(p-Cl–Ph)$_3$PO. ^{1}H-NMR/$CDCl_3$: δ 7.56 ppm (m), 7.61 ppm (m) and ^{31}P-NMR/$CDCl_3$ 29.4 ppm (neat H_3PO_4 as external reference). MS (EI): *m/e* 380 (^{35}Cl), 382 (^{37}Cl); (CI, ammonia) 381 (^{35}Cl), 383 (^{37}Cl) ($M+H^+$); 398 (^{35}Cl), 400 (^{37}Cl) ($M+NH_4^+$).

4.3. General procedure for deoxygenation of epoxides

The epoxide (30 mmol) and MTO (3 mmol) were dissolved in 100 ml benzene. A triphenylphosphine (31 mmol in 100 ml benzene) was added dropwise over 6 h with stirring under Ar, which was continued for another 12 h. The olefins were isolated by distillation under reduced pressure. The products were identified spectroscopically.

Cyclododecene, ^{1}H-NMR/$CDCl_3$: δ 1.33 ppm (m), 2.06 ppm (m) 5.38 ppm (m). Unsaturated ^{13}C-NMR/$CDCl_3$ at δ 130.23 ppm and 131.78 ppm (two isomers from cis and trans isomers of the starting epoxide). GC–MS: *m/e* 166 (cyclododecene); 278 ($OPPh_3$).

Cyclohexene, ^{1}H-NMR/$CDCl_3$: δ 1.61 ppm (m), 1.99 ppm (m) 5.68 ppm (m). GC–MS:*m/e* 82 (cyclohexene); 278 ($OPPh_3$).

trans-Stilbene, ^{1}H-NMR/$CDCl_3$: δ 7.09 ppm (m), 7.22 ppm (m), 7.32 ppm (m), 7.48 ppm (m). GC–MS: *m/e* 180 (*trans*-stilbene); 278 ($OPPh_3$).

cis-Stilbene, ^{1}H-NMR/$CDCl_3$: δ 6.58 ppm (m), 7.19 ppm (m). GC–MS: *m/e* 180 (*cis*-stilbene); 278 ($OPPh_3$).

Styrene, ^{1}H-NMR/$CDCl_3$: δ 5.20 ppm (d), 5.71 ppm (d), 6.68 ppm (dd), 7.29 ppm (m). GC–MS: *m/e* 104 (styrene); 278 ($OPPh_3$).

2,3-Dimethyl-2-butene, ^{1}H-NMR/$CDCl_3$: δ 1.65 ppm (s). GC–MS: *m/e* 84 (2,3-dimethyl-2-butene); 278 ($OPPh_3$).

4.4. Deoxygenation of sulfoxides

The general procedure used for the epoxides was used.

PhSMe, ^{1}H-NMR/$CDCl_3$: δ 7.23 ppm (m), 7.12 ppm (m) and 2.44 ppm (s). ^{13}C-NMR/$CDCl_3$ of methyl is 15.84 ppm. GC–MS: *m/e* 124 (PhSMe); 278 ($OPPh_3$)

Ph_2S, ^{1}H-NMR/$CDCl_3$: δ 7.28 ppm (m). GC–MS: *m/e* 186 (Ph_2S); 278 ($OPPh_3$).

$PhSCH{=}CH_2$, ^{1}H-NMR/$CDCl_3$: δ 7.30 ppm (m), 6.54 ppm (dd) and 5.32 ppm (m). GC–MS: *m/e* 136 ($PhSCH{=}CH_2$); 278 ($OPPh_3$).

(*p*-Me–Ph)$_2$S, ^{1}H-NMR/$CDCl_3$: δ 7.11 ppm (m), 6.88 ppm (m) and 2.25 ppm (s). GC–MS: *m/e* 214 ((*p*-Me–Ph)$_2$S); 278 ($OPPh_3$).

(*n*-Bu)$_2$S, ^{1}H-NMR/$CDCl_3$: δ 2.49 ppm (q), 1.58 ppm (m), 1.40 ppm (m) and 0.92 ppm (t). GC–MS: *m/e* 146 ((*n*-Bu)$_2$S); 278 ($OPPh_3$).

(*iso*-Pr)$_2$S, ^{1}H-NMR/$CDCl_3$: δ 2.99 ppm (m) and 1.27 ppm (d). GC–MS: *m/e* 118 ((*iso*-Pr)$_2$S); 278 ($OPPh_3$).

(*p*-Cl–Ph)$_2$S, GC–MS: *m/e* 255 ((*p*-Cl–Ph)$_2$S); 278 ($OPPh_3$).

4.5. Deoxygenation of N-oxides

$PhNMe_2$. GC–MS: *m/e* 121 (2,3-dimethyl-2-butene); 278 ($OPPh_3$) and likewise the other tertiary amines formed from the *N*-oxides. *p*-Me–$PhNMe_2$, 135; *p*-F–$PhNMe_2$, 139; *p*-Br–$PhNMe_2$, 200; *p*-NO_2–$PhNMe_2$, 166 and pyridine, 79.

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