Table I. Reaction of 2 or 4 with Aniline

Solvent (bp, °C)	Reaction time, h	Reaction of 2 yield, %		Reaction of 4 yield, %	
		5	1	5	3
Dioxane (101)	4	96	90	89	54
Benzene (80)	4	85	86	98	45
Acetone (56)	4	84	90	89	36
Dichloromethane (40)	10	42	*a*	73	*b*

[a] A mixture of 1 and unreacted 2 was obtained. [b] A mixture of 3 and unreacted 4 was obtained.

conditions, **5** was isolated in 5% yield after 48 h. The reaction was then carried out in dioxane, benzene, acetone, and dichloromethane at their boiling temperatures, and the products, diphenylurea (**5**) and *N*-hydroxyphthalimide (**1**), were isolated by fractional recrystallization. Table I summarizes the results of the reactions in the four solvents. Compound **4** was similarly treated with aniline, and the results are included in Table I. As shown in the table, **5** was isolated in good yield from **2** in dioxane, benzene, or acetone despite the small scale reaction. When dichloromethane was used as a solvent, however, the yield of **5** was much lower and a mixture of **1** and unreacted **2** was obtained, presumably because of the low boiling temperature of the solvent. Compound **4** also gave **5** in good yield, even in dichloromethane, which suggests that **4** is more reactive toward aniline than **2**.

The reactivity of **2** and **4** was then compared by the reaction of equimolar amounts of **2**, **4**, and aniline. After repeated fractional recrystallization, the expected five compounds were isolated in yields as follows: **5**, 96%; **1**, 39%; **3**, 30%; **2**, 48%; and **4**, 30%. The fact that more **2** was recovered than **4** seems to support the idea that **4** is more reactive than **2**, though the isolated amount of **3** is less than that of **1** due to the difficulty in recrystallization of **3**.[9] This is in good accordance with the tendency of a compound with a more acidic leaving component to show better reactivity in the amide-forming nucleophilic substitution reaction;[10] pK_a values of **1** and **3** are 7.0[11] and 6.0,[12] respectively. Although both **2** and **4** were thus shown to be good isocyanate generators, **2** was considered to be a better one in terms of the ease in recovering the starting *N*-hydroxy compound.

Experimental Section

Melting points were determined on a Mel-Temp apparatus and are uncorrected. IR spectra were recorded on a Jasco IR-G spectrometer. NMR spectra were obtained on Jeol JNM-MH-60 or Hitachi R-24 spectrometers. Elemental analyses were performed by Shonan Bunseki Center, Kanagawa, Japan.

Materials. *N*-Hydroxyphthalimide (**1**) was prepared according to the procedure reported by Mazur and Plume.[13] *N*-Hydroxysuccinimide (**3**) was synthesized by the method of Anderson, Zimmerman, and Callahan.[14] All of the solvents that were used were dried by the usual manner.

Phthalimido Phenylcarbamate (2). To a solution of 1.14 g (7 mmol) of **1** in 20 mL of dry dioxane was added 0.76 mL (0.83 g, 7 mmol) of phenyl isocyanate and then a drop of dibutyltin dilaurate as catalyst. The solution was stirred at room temperature, and precipitation began to take place in 4 h. The stirring was discontinued after 6 h, and the solvent was removed under reduced pressure. The resulting crystalline solid was recrystallized from dichloromethane–petroleum ether to give 1.86 g (94%) of colorless granular crystals. On rapid heating, **2** melted at 175–178 °C: IR (KBr) 3240, 1775, and 1735 cm^{-1}; NMR (Me$_2$SO-d_6) δ 6.90–7.50 (m, 5, C_6H_5–N), 7.75–7.90 (m, 4, $C_6H_4(CO)_2N$), and 8.45 (broad s, 1, NH). Anal. Calcd for $C_{15}H_{10}N_2O_4$: C, 63.83; H, 3.57; N, 9.93. Found: C, 64.22; H, 3.80; N, 9.68.

Succinimido Phenylcarbamate (4). Starting from 0.806 g (7 mmol) of *N*-hydroxysuccinimide and 0.75 mL (0.83 g, 7 mmol) of phenyl isocyanate, 1.41 g (86%) of **4** was obtained as colorless plates after recrystallization from chloroform–petroleum ether. On rapid heating, **4** melted at 149–154 °C; IR (KBr) 3240, 1775, and 1715 cm^{-1}; NMR (Me$_2$SO-d_6) δ 2.75 (s, 4, CH_2CH_2), 6.93–7.43 (m, 5, C_6H_5), and 7.45 (broad s, 1, NH). Anal. Calcd for $C_{11}H_{10}N_2O_4$: C, 56.41; H, 4.30; N, 11.96. Found: C, 56.12; H, 4.20; N, 12.13.

Reaction of 2 and Aniline. To a solution of 0.846 g (3 mmol) of **2** in 8 mL of dry acetone was added 0.285 mL (0.29 g, 3.1 mmol) of aniline. The solution was heated at reflux for 4 h and evaporated under reduced pressure to give a slightly yellow solid. It was washed with petroleum ether to remove the unreacted aniline and then recrystallized from dioxane to give 0.51 g of **5** as colorless needles. The filtrate was concentrated under reduced pressure, and the residual solid was fractionally recrystallized from dioxane–petroleum ether to give an additional 0.05 g of **5**, 0.08 g of a mixture of **5** and **1**, and 0.44 g (90%) of **1**. The total yield of **5** was 0.56 g (84%), mp 241–242 °C (lit.[15] mp 241–242 °C).

Succinimide derivative **4** was treated with aniline in the same way. The dried mixture was washed with water to remove **3**, which was recrystallized from ethyl acetate.

Reaction of a Mixture of 2 and 4 with Aniline. To a solution of 0.846 g (3 mmol) of **2** and 0.703 g (3 mmol) of **4** in 8 mL of dry acetone was added 0.273 mL (0.279 g, 3 mmol) of aniline. After heating the solution for 4 h, the solvent was removed under reduced pressure. The residual solid was fractionally recrystallized from chloroform–hexane to give 0.608 g (96%) of **5**, 0.189 g (39%) of **1**, 0.105 g (30%) of **3**, 0.403 g (48%) of **2**, and 0.211 g (30%) of **4**.

Registry No.—**1**, 524-38-9; **2**, 60506-34-5; **3**, 6066-82-6; **4**, 23583-11-1; **5**, 102-07-8; phenyl isocyanate, 103-71-9; aniline, 62-53-3.

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Oxidative Acetoxylation of Anisole by Ceric Ammonium Nitrate in Acetic Acid[1]

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Substitution of hydrogen atoms in aromatic compounds by acyloxy groups via oxidative routes is being given considerable attention in view of the mechanistic implication as well as of the synthetic potential.[2,3]

We have shown that ceric ammonium nitrate (CAN) in acetic acid is a suitable reagent for the functionalization of polymethylbenzenes.[4] Substitution of benzylic C–H bonds was the only reaction observed with all the substrates investigated, with the sole exception of mesitylene, for which nu-

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Table I. Kinetic Data for the Reaction of Anisole with CAN in Acetic Acid at 40 °C[a]

[Anisole], M	[CAN], ×10^4 M	$t_{5\%}$ × [anisole], s M[b]	[NH_4NO_3], ×10^2 M	[$Ce^{III}(NO_3)_3$], ×10^4 M
0.101	12.9	75 ± 10		
0.0203	12.9	74 ± 5		
0.0101	12.9	79 ± 5		
0.103	12.9	28 ± 2	2.02	
0.102	1.26	25 ± 2	2.02	
0.104	12.9	410 ± 70	2.02	16.2
0.104	6.44	440 ± 50	2.02	16.2
0.104	3.22	440 ± 50	2.02	16.2
0.104	1.29	490 ± 70	2.02	16.2
0.111	1.29	155 ± 10	2.02	4.05
0.114	1.29	790 ± 100	2.02	29.7

[a] In the absence of light and of oxygen. [b] Time at 5% of reaction ($t_{5\%}$) multiplied by the concentration of anisole. Each value is the average of three determinations.

Table II. Isomer Distribution in Different Oxidative Acetoxylation Reactions of Anisole

Oxidizing system	Isomer distribution, % Ortho	Meta	Para	Ortho/para ratio
CAN, AcOH[a]	45		55	0.82
$Ag(bpy)_2S_2O_8$, AcOH, AcONa 0.5 M[b]	68	1	31	2.19
Anodic oxidation, AcOH, AcONa 1.0 M[c]	67.4	3.5	29.1	2.31
AcONa 0.3 M	57.3	3.2	39.5	1.45
AcONa 0.1 M	60.2	2.2	37.6	1.60
$Pb(OAc)_4$, AcOH[d]	18		82	0.22

[a] This work. [b] Reference 13. [c] Reference 14. [d] Reference 15 (a 5:95 ortho/para ratio is obtained when the reaction is carried out in the presence of air.

clear acetoxylation also occurred as a side reaction. We now wish to report that with anisole, i.e., an electron-rich aromatic substrate bearing no benzylic C–H bonds, CAN in AcOH smoothly promotes nuclear acetoxylation.

Results and Discussion

A homogeneous solution in AcOH of anisole and CAN was kept at 40 °C under nitrogen and in the dark for 22 h. VPC analysis of the crude product showed the presence of *o*- and *p*-acetoxyanisole whereas no peak attributable to the meta isomer was observed. The two acetoxyanisoles were also isolated in a pure form by means of preparative VPC. In order to allow for the possible further oxidation of initially formed reaction products,[5] the isomer distribution was determined by VPC at different times in the early stages of the reaction. It was found that the ortho/para ratio remains unchanged up to ca. 30% reaction, the average value being 0.81 ± 0.03. Thus the ortho/para ratio appears unaffected by further oxidation of the two isomers.

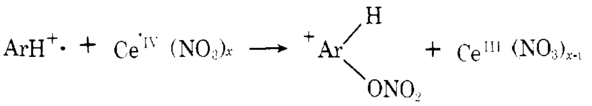
The determination of the times at 5% of reaction revealed that the reaction is approximately first order in anisole and in CAN[7] and has an order −1 in Ce(III). The experiments in which the concentrations of Ce(IV) or Ce(III) were changed were generally carried out in the presence of an excess of NH_4NO_3 in order to keep the total salt concentration practically constant. The addition of NH_4NO_3, which was the salt of choice since its anion is also the ligand in the oxidizing complex, exerts a significant acceleration of the reaction rate as already observed in other oxidation reactions by CAN.[4a,8] All kinetic results are reported in Table I. The results, together with the well-known ability of Ce(IV) compounds to act as one-electron transfer reagents toward electron-rich aromatic compounds,[10] allow us to suggest that the reaction of Ce(IV) with anisole leads to the formation, in a fast and reversible step, of a radical cation which then slowly decomposes to products (eq 1).

$$ArH + Ce^{IV} \rightleftharpoons ArH^{+}\cdot + Ce^{III} \qquad (1a)$$

$$ArH^{+}\cdot \xrightarrow[\text{more than one step}]{\text{slow}} \text{products} \qquad (1b)$$

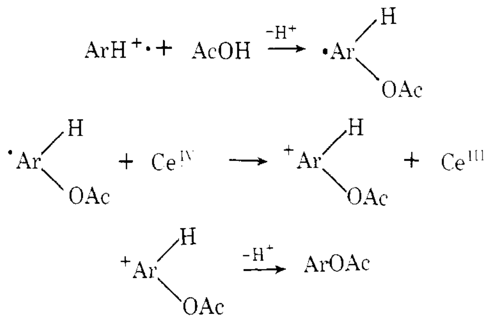
This suggestion is also supported by the fact that our kinetic pattern is practically identical to that observed in the Mn(III) induced acetoxylation of 1-methoxynaphthalene, for which a mechanism similar to that reported in eq 1 has been proposed.[11]

As to the rate-limiting transformation of the radical cation into products, a reaction with either a nucleophile or a radical (either as a free species or bound to a carrier) could in principle be envisaged. However, the observation of a first-order reaction in Ce(IV) and the absence of aryl nitrates and/or decomposition products possibly derived therefrom[12] rules out the operation of a ligand transfer reaction (eq 2)



$$ArH^{+}\cdot + Ce^{IV}(NO_3)_x \longrightarrow {}^{+}Ar\begin{smallmatrix}H\\ONO_2\end{smallmatrix} + Ce^{III}(NO_3)_{x-1} \qquad (2)$$

and strongly suggests the intervention of the mechanism of eq 3



$$ArH^{+}\cdot + AcOH \xrightarrow{-H^{+}} \cdot Ar\begin{smallmatrix}H\\OAc\end{smallmatrix} \qquad (3a)$$

$$\cdot Ar\begin{smallmatrix}H\\OAc\end{smallmatrix} + Ce^{IV} \longrightarrow {}^{+}Ar\begin{smallmatrix}H\\OAc\end{smallmatrix} + Ce^{III} \qquad (3b)$$

$${}^{+}Ar\begin{smallmatrix}H\\OAc\end{smallmatrix} \xrightarrow{-H^{+}} ArOAc \qquad (3c)$$

where a nucleophilic attack by the solvent AcOH affords a radical intermediate, which in turn can be easily oxidized by Ce(IV) to a cationic σ complex.

Interestingly, the isomer distribution in the oxidation of anisole by CAN/AcOH appears to follow the odd-electron density distribution at the various positions of the anisole radical cation.[10] The positional selectivity is higher than that reported for related oxidative acetoxylation reactions of anisole (see Table II, lines 2–5) for which, however, without a kinetic support, a nucleophilic attack of acetate ion on the radical cation has been suggested. In the latter reactions appreciable amounts of the meta isomer are formed, and the ortho/para ratio approaches the statistical value of 2. The different behavior can be accounted for by operation of the reactivity–selectivity principle, according to which the neutral, less-reactive, nucleophile AcOH is expected to exhibit increased selectivity as compared with the stronger nucleophile AcO^-. A similar view has been presented to account for different ortho/para ratios in a comparison of cyanation, acetoxylation, and trifluoroacetoxylation of the chlorobenzene radical cation.[16]

The isomer distribution of the reaction promoted by CAN is also significantly different from that exhibited in the reactions induced by $Pb(OAc)_4$ in AcOH (Table II, line 6). However, in the latter case the products of nuclear acetoxylation have been suggested to derive from the collapse of the radical cation and the associated lead species.

Experimental Section

Proton magnetic resonance spectra were taken on a Jeol JNM-C60HL spectrometer, using Me_4Si as the internal standard. Infrared spectra were obtained on a Perkin-Elmer 257 from 2% solutions in CCl_4. VPC analyses were performed on a GI Fractovap (C. Erba). UV spectra and kinetics were recorded with a Beckman DBGT spectrophotometer.

Materials. Ceric ammonium nitrate [$(NH_4)_2Ce(NO_3)_6$] (Schuchardt, 99.9% pure) was dried at 85 °C for 1 h. Acetic acid (C. Erba 99.8% pure) was thoroughly fluxed with pure nitrogen before use. Ammonium nitrate (C. Erba, 99% pure), cerous nitrate [$Ce(NO_3)_3 \cdot 6H_2O$] (C. Erba, 98% pure), anisole (C. Erba, 99% pure), *m*-methoxyphenol (Merck, 97% pure), and *p*-methoxyphenol (C. Erba, 99% pure) were commercial samples and were used as received. *o*-Methoxyphenol (Farmitalia) was distilled before use.

Methoxyphenyl Acetates. The three acetate isomers were prepared by acetylation of the corresponding phenol with acetic anhydride and acqueous alkali. Complete resolution of a mixture of the three isomers was achieved by VPC on a 1-m column, packed with 10% LAC 728, operating at 110 °C.

The Oxidation of Anisole with CAN. In a typical experiment, anisole (9.2 mmol) in 50 mL of oxygen-free acetic acid was added to a homogeneous solution of CAN (4.6 mmol) in 200 mL of the same solvent. The mixture was kept at 40 °C under nitrogen in a dark place. After 22 h (75% of Ce(IV) consumed) the reaction mixture was poured into cold ethyl ether and washed with water. After removing the solvent, the residue, which showed strong carbonyl absorption at 1770 cm^{-1}, was analyzed by VPC. Comparison of the gas chromatogram with those of authentic samples of the three isomeric acetoxyanisoles showed that *o*- and *p*-acetoxyanisole accounted for more than 95% of the reaction products, as based on peak areas. The two acetoxyanisoles were also isolated from a product mixture by means of preparative VPC on a 2-m column packed with SE 30 10% operating at 100 °C and compared with the authentic samples. No peak attributable to the meta isomer was present in the gas chromatogram thus indicating that this isomer, if present, is less than 0.5%.

Kinetic Measurements. The rates of oxidation of anisole were measured by following the disappearance of cerium(IV) in a thermostated cell compartment of a UV spectrophotometer. The optical densities were determined at 410 nm (ϵ 6.2 × 10^2 M^{-1} cm^{-1}) and in the presence of NH_4NO_3 at 360 (ϵ 4.1 × 10^3 M^{-1} cm^{-1}), 390 (ϵ 14.9 × 10^2 M^{-1} cm^{-1}), and 410 nm (ϵ 7.5 × 10^2 M^{-1} cm^{-1}) depending on the concentration of CAN.

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Registry No.—CAN, 16774-21-3; anisole, 100-66-3; *o*-acetoxyanisole, 613-70-7; *m*-acetoxyanisole, 5451-83-2; *p*-acetoxyanisole, 1200-06-2; acetic acid, 64-19-7.

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(8) Other than by a salt effect, expected for a reaction involving charged species, NH_4NO_3 might influence the oxidation rate also by affecting the equilibrium $(NH_4)_2Ce(NO_3)_6 + 2AcOH \rightleftharpoons Ce(NO_3)_4(AcOH)_2 + 2NH_4NO_3$.[9]

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A New, Mild Method for the Synthesis of Azo Compounds

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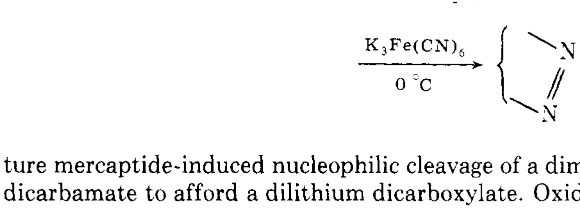
Azo compounds have long played a significant role in the development of mechanistic[1] and synthetic[2] organic chemistry. Often, however, their synthesis is encumbered by the harsh conditions of their genesis. Most methods involve the conversion of a dicarbamate to an hydrazo compound followed by oxidation to afford the desired azo linkage. While alkaline saponification of the dicarbamate at temperatures exceeding 80 °C has frequently been used,[3] in many cases only the most robust compounds survive these harsh conditions. The reductive cleavage of bis(2,2,2-trichloroethyl) esters,[4] the hydrogenolysis of benzyl esters,[5] and the β-elimination of β-tosylethyl esters[6] represent mild alternatives to alkaline saponification. Still, we have uncovered examples where even the conditions of these milder methods have proven to be incompatible with the survival of the desired product.[7]

We now wish to report that we have discovered a mild, one-pot, two-step sequence to effect the conversion of a dicarbamate to an azo linkage.[8] The conversion is effected at or below room temperature and in yields ranging from 60 to 80%; the often-times unstable hydrazo compound which is produced in most methods is bypassed entirely. The low temperatures required to effect the sequence make it possible to isolate even thermally labile azo compounds.

The method, outlined in eq 1, involves the room temperature mercaptide-induced nucleophilic cleavage of a dimethyl dicarbamate to afford a dilithium dicarboxylate. Oxidation with aqueous potassium ferricyanide at 0 °C results in the immediate evolution of gas (CO_2) and the formation of yellow Fe(II) salts.[9,10] Both the lithium salts of *n*-propyl mercaptan and methyl mercaptan have been successfully utilized. These reagents, 0.5–1.3 M in HMPA, are easily prepared and can be stored in the refrigerator for at least 1 month without showing



(1)