product was purified by reprecipitating from methylene chloride with ether, giving 5-methylthianthreniumyl tetrafluoroborate (eq 2, R = Me): mp 194–196 °C dec; ^{1}H NMR ($CDCl_3$–CD_3CN) δ 8.22 (d, 2 H, J = 7.6 Hz), 7.69–7.92 (m, 6 H), 3.25 (s, 3 H, CH_3). Anal. Calcd for $C_{13}H_{11}S_2BF_4$: C, 49.1; H, 3.46; S, 20.1. Found: C, 49.2; H, 3.46; S, 20.5.

Reaction of $Th^{\cdot+}BF_4^-$ with Diphenylmercury. Reaction was carried out by adding 10 mL of acetonitrile to a mixture of 610 mg (2.01 mmol) of $TH^{\cdot+}BF_4^-$ and 370 mg (1.05 mmol) of diphenylmercury. Workup as described for reaction with Me_2Hg gave 327 mg (0.86 mmol, 86%) of 5-phenylthianthreniumyl tetrafluoroborate (eq 2, R = Ph), mp 244–246 °C, after reprecipitation: ^{1}H NMR ($CDCl_3$–CD_3CN) δ 8.39 (d, 2 H), 7.90 (m, 6 H), 7.49 (m, 3 H), 7.09 (m, 2 H). Anal. Calcd for $C_{18}H_{13}S_2BF_4$: C, 56.8; H, 3.42; S, 16.8. Found: C, 56.8; H, 3.45; S, 17.7.

Acknowledgment. We thank the National Science Foundation, Grant No. 86-12031, for support.

Registry No. $Th^{\cdot+}BF_4^-$, 60896-34-6; $NOBF_4$, 14635-75-7; Th, 92-85-3; ThO, 2362-50-7; Me_2Hg, 593-74-8; Ph_2Hg, 587-85-9; 5-methylthianthreniumyl tetrafluoroborate, 32593-00-3; 5-phenylthianthreniumyl tetrafluoroborate, 32593-01-4.

Preparation of 2-Aryladamantanes and 3-Aryldiamantanes by Improved Ionic Hydrogenation of the Corresponding Tertiary Alcohols with Sodium Borohydride–Triflic Acid or Formic Acid–Triflic Acid[1]

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Received April 12, 1988

Introduction

In the expanding chemistry of hydrocarbon derivatives of adamantane and adamantane analogues, the preparation of tertiary derivatives is accomplished more readily than that of secondary derivatives.[2,3] The number of secondary substituted adamantanes is limited since their synthesis could formerly be achieved only by ring-closure reactions in low yield.[2] While preparative procedures for certain secondary derivatives of adamantane are available,[3] many have not yet been prepared.

Preparation of several tertiary alkyl and aryl derivatives (e.g., methyl, ethyl, benzyl) of adamantane has been reported by Schleyer et al. by a Grignard coupling method under rigorous conditions.[4] The method was also found to be useful in the preparation of other alkyl and aryl tertiary derivatives of adamantanes.[5] The Grignard and the organolithium coupling method were found to be ineffective for the preparation of alkyl and aryl secondary derivatives of adamantane and diamantane.[6]

Preparation of 2-phenyladamantane and isomeric 2-tolyladamantanes was reported in 50–70% yield by Wynberg et al. using dehydroadamantane (tetracyclo[3.3.1.1^{3,7}.0^{2,4}]decane) and $AlCl_3$ or BF_3·OEt_2 in benzene and toluene, respectively.[7] The method is involved since the synthesis of the precursor dehydroadamantane is a multistep process.[8] We now report an efficient method for the preparation of 2- and 3-aryl derivatives of adamantane and diamantane, respectively, using improved ionic hydrogenation of the corresponding tertiary alcohols.

Table I. Percent Yield of 2-Aryladamantanes and 3-Aryldiamantanes Obtained by $NaBH_4$ Reduction of Aryladamantanols (Diamantanols)

2-aryladamantanes and 3-aryldiamantanes	% yield (isolated) $NaBH_4$–CF_3COOH	% yield (isolated) $NaBH_4$–CF_3SO_3H	% yield (isolated) HCO_2H–CF_3SO_3H	mp, °C (bp)
1a	81	98	94	30–31
1b	72	95	94	57–58
1c	70	96	94	(120–121 [1.2 Torr])
1d	74	99	98	58–59
2a	80	97	94	73–74
2b	70	94	95	76–77
2c	77	98	95	56–57
2d	75	99	96	87–88

Results and Discussion

The reduction of different functional groups with sodium borohydride in carboxylic acids has been used over the years.[9] Thus, $NaBH_4$ in neat carboxylic acid media sequentially reduces and alkylates N-heterocycles to give the corresponding *N*-alkyl compounds.[10] The reagent combination was further used for the alkylation of amines,[10a,11] reduction of oximes,[12] nitrimine,[13] amide,[14] and nitrile.[15] Diaryl ketones and di- and triarylmethyl alcohols were found to give corresponding hydrocarbons in high yield with $NaBH_4$–CF_3COOH.[16,17] Under certain conditions, this reagent system was found to convert arenes to 1,1,1-trifluoro-2,2-diarylethanes in moderate yield.[18] In the case of arylalkylmethyl alcohols only partial reduction to hydrocarbons was observed. Thus, in the reduction of 2-phenyl-2-propanol with $NaBH_4$–CF_3COOH, only 45%

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Table II. GC-MS Data

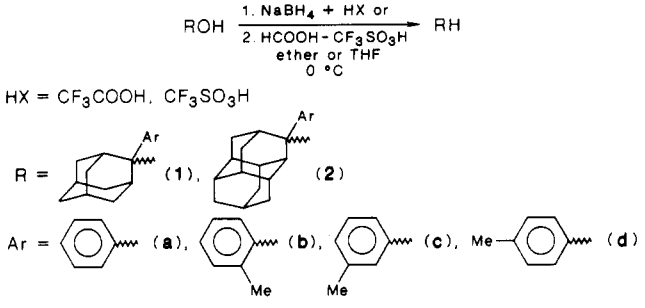
compd	MS data of 2-aryladamantanes and 3-aryldiamantanes
1a	212 (M^+, 86), 213 (M + 1, 14), 121 (M - Ph, 33), 117 (16), 115 (26), 105 (18), 93 (36), 91 (89), 79 (100), 77 (34)
1b	226 (M^+, 100), 211 (M - CH_3, 89), 129 (28), 121 (M - Ph - CH_3, 25), 117 (16), 115 (27), 105 (70), 91 (62), 79 (100), 77 (38)
1c	226 (M^+, 100), 211 (M - CH_3, 66), 129 (23), 121 (M - Ph - CH_3, 20), 115 (21), 105 (62), 91 (58), 79 (76), 77 (36)
1d	226 (M^+, 100), 211 (M - CH_3, 99), 129 (31), 121 (M - Ph - CH_3, 19), 115 (27), 105 (94), 91 (80), 79 (93), 77 (43)
2a	264 (M^+, 72), 265 (M + 1, 15), 187 (2), 173 (15), 129 (21), 117 (27), 94 (60), 91 (M - dia, 100), 79 (46)
2b	278 (M^+, 51), 279 (M + 1, 12), 263 (M - CH_3, 51), 173 (11), 129 (30), 117 (29), 105 (74), 91 (M - dia, 100), 79 (57)
2c	278 (M^+, 74), 279 (M + 1, 16), 263 (M - CH_3, 55), 173 (11), 129 (30), 117 (27), 105 (78), 91 (M - dia, 100), 79 (60)
2d	278 (M^+, 99), 279 (M + 1, 22), 263 (M - CH_3, 79), 173 (10), 129 (28), 117 (28), 105 (88), 91 (M - dia, 100), 79 (59)

Table III. ^{13}C NMR Data

compd	^{13}C chemical shifts		
	cage carbons	aromatic carbons	others
1a	C_5 28.05 (d), C_7 28.29 (d), $C_1 = C_3$ 31.27 (d), $C_8 = C_{10}$ 31.10 (t), C_6 38.10 (t), $C_4 = C_9$ 39.36 (t), C_2 47.0 (d)	125.22 (d), 126.87 (d), 128.20 (d), 144.03 (s)	
1b (ortho)	C_5 27.64 (d), C_7 28.24 (d), $C_1 = C_3$ 31.84 (d), $C_8 = C_{10}$ 32.74 (t), C_6 38.13 (t), $C_4 = C_9$ 40.40 (t), C_2 46.57 (d)	125.36 (d), 126.60 (d), 130.76 (d), 136.47 (s), 144.05 (s)	19.80 (q, CH_3)
1c (meta)	C_5 27.90 (d), C_7 28.12 (d), $C_1 = C_3$ 31.13 (d), $C_8 = C_{10}$ 32.05 (t), C_6 37.96 (t), $C_4 = C_9$ 39.25 (t), C_2 46.83 (d)	123.79 (d), 125.85 (d), 127.63 (d), 127.97 (d), 137.39 (s), 144.30 (s)	21.64 (q, CH_3)
1d (para)	C_5 27.87 (d), C_7 28.11 (d), $C_1 = C_3$ 31.10 (d), $C_8 = C_{10}$ 31.98 (t), C_6 37.98 (t), $C_4 = C_9$ 39.21 (t), C_2 46.55 (d)	126.70 (d), 128.84 (d), 134.46 (s), 141.33 (s)	20.86 (q, CH_3)
2a	47.96 (d), 40.59 (d), 39.56 (t), 39.02 (d), 38.45 (t), 38.07 (t), 38.00 (t), 37.59 (d), 37.40 (d), 37.09 (d), 33.00 (t), 32.08 (d), 28.76 (d), 26.28 (d)	125.10 (d), 126.94 (d), 128.09 (d), 143.98 (s)	
2b (ortho)	48.01 (d), 41.29 (d), 40.79 (t), 40.14 (d), 38.54 (t), 38.07 (t), 38.07 (t), 37.66 (d), 37.48 (d), 36.99 (d), 34.02 (t), 33.02 (d), 29.65 (d), 26.26 (d)	143.79 (s), 136.63 (s), 130.82 (d), 126.82 (d), 125.34 (d), 125.29 (d)	19.98 (q, CH_3)
2c (meta)	47.97 (d), 41.25 (d), 40.75 (t), 40.10 (d), 38.50 (t), 38.01 (t), 38.01 (t), 37.61 (d), 37.44 (d), 36.94 (d), 33.99 (t), 32.98 (d), 29.60 (d), 26.23 (d)	143.76 (s), 136.62 (s), 130.78 (d), 126.79 (d), 125.32 (d)	19.96 (q, CH_3)
2d (para)	47.64 (t), 40.64 (t), 39.57 (d), 39.02 (t), 38.48 (d), 38.10 (d), 38.03 (d), 37.66 (t), 37.44 (t), 37.13 (t), 33.00 (d), 32.08 (t), 28.76 (t), 26.33 (t)	141.20 (s), 134.44 (s), 128.82 (d), 126.82 (d)	20.87 (q, CH_3)

cumene could be obtained.[16]

We now report the effective reduction of aryl-substituted tertiary adamantanols and diamantanols with $NaBH_4$–trifluoromethanesulfonic (triflic) acid. Triflic acid was added dropwise to a mixture of 2-aryl-2-adamantanols and $NaBH_4$ in diethyl ether at 0 °C. After being stirred for 10 min, the reaction mixture was worked up; 2-aryladamantanes were obtained in near-quantitative yield. The procedure when applied to 3-aryl-3-diamantanols gave the corresponding hydrocarbons also in near-quantitative yield.



For comparison, we also used the $NaBH_4$–CF_3COOH system, which was found to be an efficient reagent for the reduction of certain diarylmethyl alcohols.[16] The results as summarized in Table I show that reduction of the aryl-substituted polycyclic alcohols to the corresponding hydrocarbons with $NaBH_4$–CF_3SO_3H is more effective than with the $NaBH_4$–CF_3COOH system, although the latter gives higher yields with studied tertiary adamantanols and diamantanols as with 2-phenyl-2-propanol.[16]

Reduction of triphenylmethyl alcohols to triphenylmethane with formic acid is known,[19] although the reaction is slow at room temperature (after 2 weeks, a yield of 91% was obtained). We now find, however, that the reduction of triphenylmethyl alcohol in HCOOH is extremely effective in the presence of a catalytic amount of triflic acid, to give in only 10 min a 97% yield. Application of the HCOOH–CF_3SO_3H system to the reduction of tertiary aryl polycycloalkyl alcohols gave near-quantitative yields of the corresponding hydrocarbons (Table I). Both the $NaBH_4$–CF_3SO_3H and HCOOH–CF_3SO_3H systems are found to be more effective reducing systems than $NaBH_4$–CF_3COOH, indicating the advantages of using superacidic triflic acid in the ionic hydrogenations.

The presently developed procedure is an excellent method of preparing secondary aryl derivatives of adamantane and diamantane and can be generally applied for the preparation of similar derivatives of other polycyclic systems.

Experimental Section

Sodium borohydride, 2-adamantanone, bromobenzene, and isomeric bromotoluenes were available from Aldrich. Trifluoroacetic acid (Aldrich) and trifluoromethanesulfonic acid (3M Co.) were distilled prior to use. Diethyl ether and THF were dried over sodium through reflux. 3-Diamantanone was prepared according to a literature procedure.[20]

GC analysis was carried out on a Varian gas chromatograph (Model 3700) using a 30-m capillary column (quartz silica, DB-1). GC–MS analysis was carried out on a Finnigan Mat Model 700 GC–MS equipped with an ion-trap detector and interfaced with a Varian Associates Model 3500 gas chromatograph. ^{13}C NMR spectra were recorded on a VXR-200 superconducting NMR instrument. Melting points were determined on a Mettler apparatus and are uncorrected.

General Method of Preparation of 2-Aryl-2-adamantanols (3-Aryl-3-diamantanols). To a stirred solution of 12 mmol of

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ketone (2-adamantanone or 3-diamantanone) in dry ether at 0 °C was added dropwise 15 mmol of arylmagnesium bromide in ether. After the addition of Grignard reagent, the reaction mixture was stirred at room temperature for 1 h, followed by reflux for about 3 h. The reaction mixture on cooling was quenched in ice–water–HCl. Extraction into ether, drying over $MgSO_4$, and evaporation gave the tertiary alcohols in near-quantitative yields. Further purification of the alcohols was carried out on a silica gel column (ether as eluent). The purity of the alcohols was checked by GC and GC–MS.

General Method of Reduction of 2-Aryl-2-adamantyl (3-Aryl-3-diamantyl) Alcohols with $NaBH_4$. To a well-stirred mixture of 2-aryl-2-adamantanols or 3-aryl-3-diamantanols (10 mmol) and powdered $NaBH_4$ (10 mmol) in dry ether (20 mL) in a 100-mL round-bottom flask fitted with a reflux condenser was added dropwise triflic acid (50 mmol) at 0 °C over 10 min under dry nitrogen. After the addition, the reaction mixture was stirred for another $^1/_2$ h. It was then quenched in ice–water–bicarbonate. Extraction with ether, drying over $MgSO_4$, and solvent removal gave crude 2-aryladamantanes or 3-aryldiamantanes. Purification on a silica gel column (hexane as eluent) afforded pure hydrocarbon products, which subsequently were characterized by their ^{13}C NMR spectra, GC–MS, and elemental analysis.

2-Tolyladamantanes (isomeric): calcd C 90.20, H 9.80. Found: (ortho) C 90.41, H 9.86; (meta) C 90.28, H 9.80; (para) C 90.45, H 9.60.

3-Tolyldiamantanes (isomeric): calcd C 90.59, H 9.41. Found: (ortho) C 90.39, H 9.29; (meta) C 90.57, H 9.75; (para) C 90.63, H 9.57.

3-Phenyldiamantane: calcd C 90.85, H 9.15. Found: C 90.81, H 9.10.

General Method of Reduction with HCO_2H–CF_3SO_3H. To an ice-cold solution of 2-aryl-2-adamantanols or 3-aryl-3-diamantanols (10 mmol) in dry ether (20 mL) was added triflic acid (ca. 1–2 mmol), followed by dropwise addition of formic acid (96%, 12 mmol) over 10 min with stirring under dry nitrogen. The ice–cold bath was then removed, and the reaction was continued for another 5–10 min. It was then quenched in ice–bicarbonate and extracted with ether. The ethereal layer was dried over $MgSO_4$, filtered, and evaporated to give crude 2-aryladamantanes or 3-aryldiamantanes. Column chromatography on silica gel (hexane eluent) afforded pure hydrocarbon products.

Acknowledgment. Support of our work by the National Institutes of Health is gratefully acknowledged.

Registry No. **1a**, 19066-24-1; **1a**-OH, 29480-18-0; **1b**, 115942-81-9; **1b**-OH, 115942-77-3; **1c**, 19214-04-1; **1c**-OH, 76481-45-3; **1d**, 19066-25-2; **1d**-OH, 29480-17-9; **2a**, 115942-82-0; **2a**-OH, 95531-42-3; **2b**, 115942-83-1; **2b**-OH, 115942-78-4; **2c**, 115942-84-2; **2c**-OH, 115942-79-5; **2d**, 115942-85-3; **2d**-OH, 115942-80-8; PhBr, 108-86-1; *o*-MeC_6H_4Br, 95-46-5; *m*-MeC_6H_4Br, 591-17-3; *p*-MeC_6H_4Br, 106-38-7; 2-adamantanone, 700-58-3; 3-diamantanone, 30545-23-4.

Hydrogen-Bonding Basicity of 1-Methyl-2-pyridone and of the Nitrogen Atom of Pyridine Derivatives toward Imides[1]

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Received March 31, 1988

We have previously studied equilibria in the hydrogen bonding of N–H acids, the only kind of acids involved in hydrogen bonding between the two strands of DNA, with monocoordinate oxygen bases, one of the two kinds of bases involved in such hydrogen bonding.[2] The bases studied were sulfoxides, a phosphoroxy base, and a saturated cyclic amide; we have now studied 1-methyl-2-pyridone, whose basic functional group is more nearly identical with those in DNA. We have also studied hydrogen bonding of N–H acids to the nitrogen atom of pyridine derivatives, the only other kind of hydrogen bonding holding the two strands of DNA together.

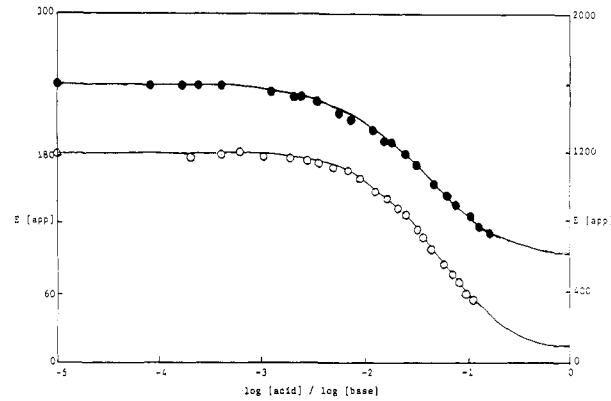

Figure 1. Plots of apparent extinction coefficients vs the logarithm of the concentration of the nonabsorbing reagent in the interaction of 2-methyl-2-ethylsuccinimide and 1-methyl-2-pyridone in carbon tetrachloride at 25 °C. Open circles are for the imide and refer to the scale on the left margin. Solid circles are for methylpyridone and refer to the scale on the right margin.

Experimental Section and Data Treatment

The 1-methyl-2-pyridone used was redistilled just before use to remove the pink color that is formed on standing. The sources and properties of the imides[2] and pyridines[3] have been described as have the techniques used for the UV and IR measurements and the methods used to calculate the equilibrium constants. Allowance was made for the dimerization of the imides and the further polymerization of 2-methyl-2-ethylsuccinimide.[2] In all cases the sum of the squares of the deviations from the observed absorbances was minimized except in the case of 2-aminopyridine where the sum of the squares of the deviations from the observed apparent extinction coefficients was minimized. The concentration of the absorbing reactant was changed, by no more than 55%, by addition of the other reactant, whose concentration ranged from zero to that required to give the maximum percent conversion listed in Table I. In all cases addition of the nonabsorbing reagent caused a decrease in the apparent extinction coefficient of the absorbing reagent at the wavelength at which measurements were made. The maximum absorbance was between 0.71 and 0.98.

Figure 1 contains plots of data on 2-methyl-2-ethylsuccinimide and 1-methyl-2-pyridone; both the UV and the IR data are plotted. As shown in Table I the K values differed by no more than 30%.

The thermodynamic pK_a of 1-pentyluracil was determined by potentiometric titration by using the Davies equation to calculate the effects of ionic strength.[4]

Results and Discussion

Figure 2, which is a plot of log K for hydrogen bonding to 1-methyl-2-pyridone vs the pK_a values for the imides, shows the expected tendency for the equilibrium constants to increase with increasing acidity of the acids involved. The points describe a straight line of slope (standard deviation) 0.342 (0.014). This is essentially the same as that of a similar plot for hydrogen bonding of *N*-methylpyrrolidone to about the same set of imides.[2] Thus the heteroaromatic amide 1-methyl-2-pyridone behaves in the same manner as a saturated amide. The line in Figure 2 would presumably be applicable to any imide if the steric conditions around the acidic hydrogen atom do not differ

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