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The epimetallation and carbonation of carbonyl and imino derivatives: Epivanadation route to 2-amino and 2-hydroxy acids ☆

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Dedicated to Professor Gerhard Erker on the occassion of his 60th birthday.

Abstract

The feasibility of hydrocarboxylating carbonyl and imino derivatives by the two-step process of epimetallation and carbonation has been demonstrated with the model substrates of 9-fluorenone and 9-fluorenone anil. With lithium vanadium dihydride as the epimetallating agent, such hydrocarboxylation has led to a 75% yield of 9-hydroxy-9-fluorenecarboxylic acid and a 65% yield of 9-(*N*-phenylamino)-9-fluorenecarboxylic acid, respectively. Some initial success in extending the scope of this reaction to other substrates, such as benzophenone, has been achieved by using other epimetallating agents, like the presumed $LiV(CH_3)_2$ and $Ti(OPr^i)_2$. A brief review of the processes and organic synthetic applications of epimetallation and transfer epimetallation of C–C π-bonds is offered as background.

Keywords: Hydrocarboxylation; Epimetallation of C=E bond (E = O, N–R); Carbonation; Lithium vanadium(I) dihydride; Presumed lithium dimethylvanadate(I)

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1. Introduction

1.1. Leitmotiv of this festschrift and its relation to organic synthesis with organometallics

The theme of this special issue dedicated to Gerhard Erker, ‘‘Insertion Chemistry of the Metal–Carbon Bond’’, celebrates both Professor Erker’s prominent research contributions to this field, as well as this key reaction itself, which has continually fascinated organic chemists since the time of Frankland [1]. The studies of Frankland, Butlerov, Zelinsky, Reformatsky and their students with zinc alkyls and carbonyl derivatives [2] had set the stage for the revolutionary impact that the Grignard reagent was to have on organic synthesis [3]. Ever since, every new preparation or type of carbon–metal σ-bond has been seized upon by organic chemists, eager to learn what insertions of π-bonded substrates could be achieved. The most recent example of such stupendously successful research has been Karl Ziegler’s synthesis of unsolvated aluminum alkyls and his discoveries of the multi-insertions of olefins and acetylenes into C–Al or into the ensuing carbon–transition metal bonds [4]. From these seemingly simple studies of C–M bond insertions whole new vistas of organic synthesis and polymerization processes have unfolded.

1.2. Epimetallation of carbon–carbon π-bonds and the sigma C–M bond character of such reaction products

Over the last two decades the ubiquitous transition metal π-complexes (**3**) formed between subvalent transition salts $M_t^nL_m$ (**1**) and C=C or C≡C linkages (**2**) have come to be recognized as varying in structure from principally

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☆ Part 42 of the series, “Organic Chemistry of Subvalent Transition Metal Complexes; for the previous Part 41, see J.J. Eisch, J.N. Gitua, Organometallics 26 (2007) 778 (Ref. [13b]).

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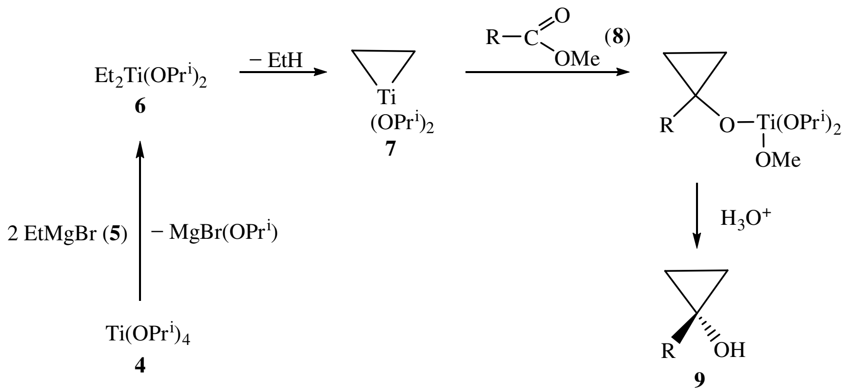
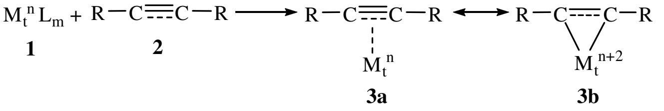


Scheme 1.

that of a π- or η^2-complex (**3a**) to that of a metallacyclopropane or -cyclopropene ring with significant σ C–M bonding and an increase of the oxidation number of M_t^n toward M_t^{n+2} (**3b**) (Eq. (1)). The relative importance of resonance structures **3a** and **3b** can be assessed in specific cases by analyzing structural information for those complexes that can be isolated or by determining the chemical properties of such complexes in solution. Such an analysis has been carried for a number of nickel, titanium, zirconium, chromium and vanadium complexes with the conclusion that the metallacyclopropane (-cyclopropene) structure (**3b**) is in better accord with the observed physical and chemical properties [5,6].



(1)

The first experimental suggestion that a metallacyclopropane could be a critical intermediate in transition metal-mediated organic synthesis was that of the Kulinkovich group in their report of an astonishing high-yielding cyclopropanol synthesis [7] (Scheme 1). The interaction of titanium(IV) isopropoxide (**4**) with two or more equivalents of EtMgBr (**5**) at −78 °C was proposed to lead to $Et_2Ti(OPr^i)_2$ (**6**), whose decomposition would provide **7**, the so-called Kulinkovich intermediate. Reaction of the two C–Ti bonds of **7** with the ester **8** would produce ultimately the cyclopropanol **9**.

The likelihood of this mechanistic path was corroborated in our group by the synthesis of the presumed intermediate **7** in an independent way [6], namely the transfer epimetallation of ethylene by $BuTi(OPr^i)_2$ with the loss of the butyl groups (**10** → **7**). (For the experimental evidence supporting the occurrence of transfer epititanations, please consult Ref. [8,9]). Proof that **7** has two distinct C–Ti bonds follows from the successive reactions of two different electrophiles, benzonitrile and CO_2, to produce upon hydrolysis keto acid **13**, logically via **11** and **12**. The final product **13** is not explicable without the intermediacy of **7** [6,10] (see Scheme 2).

Since in many of the cases of the complex formation according to Eq. (1), with TiE_2 [6], $ZrCl_2$ [11], $HfCl_2$ [11], CrCl [12], VCl [13], $Ni^0 \cdot L_n$ [14], $LiCrH_2$ [15] and $LiVH_2$ [13a], the adduct **3** has displayed the physical and chemical properties of a three-membered metallocycle **3b**, we have proposed the term of epimetallation for the reaction of **1** with **2** to produce **3b** [5]. This term has its origin and analogy with the reaction of epoxidation in organic chemistry, by which a neutral O from O_2 or a peroxyacid is added across a carbon–carbon π-bond.

1.3. Epimetallations in organic synthesis

Impressed by the fundamental importance of epititanations in the Kulinkovich cyclopropanol synthesis, we have been motivated to explore epimetallations of olefins and acetylenes with such diverse reagents as $TiCl_2$, $ZrCl_2$, $HfCl_2$, CrCl, VCl, $LiCrH_2$ and most recently $LiVH_2$, as well as their $R_2M_tE_2$ derivatives in transfer epimetallations (for reviews, consult Ref. [5,16]). The generalized metallacyclopropanes or -cyclopropenes arising from such epimetallations, **14**, have been shown to undergo an impressive array of high-yielding, often stereoselective insertions of unsaturated substrates into the strained metal–carbon σ-bond. A "palette" of useful reactions is depicted in Scheme 3 (products after hydrolysis).

1.4. Epimetallations of carbonyl and imino groups

The aforementioned success with the epimetallation and derivative formation of olefins and acetylenes has stimulated us to apply such reactions to polar π-bonds, such as C=O, C=N or C≡N groups. Our earliest attempt to realize such a process was with benzophenone (**15**) and $LiCrH_2$.[1] Treatment of **15** with $LiCrH_2$ and subsequent treatment with D_2O gave principally dideuterio **17**. Thus the main reaction was one of epichromation to produce

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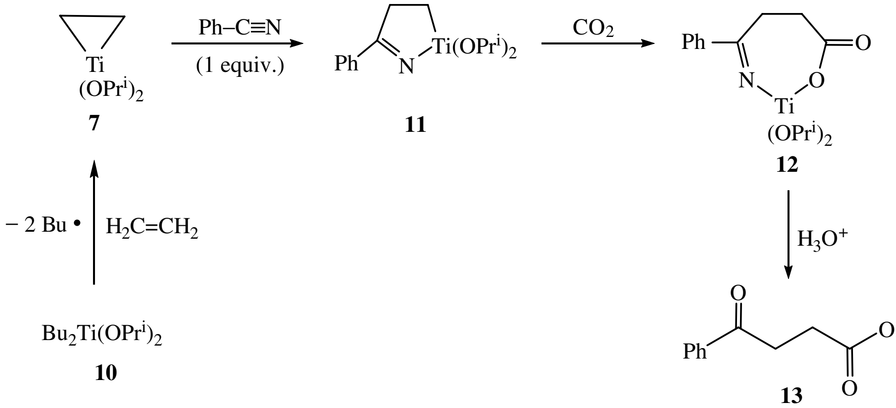
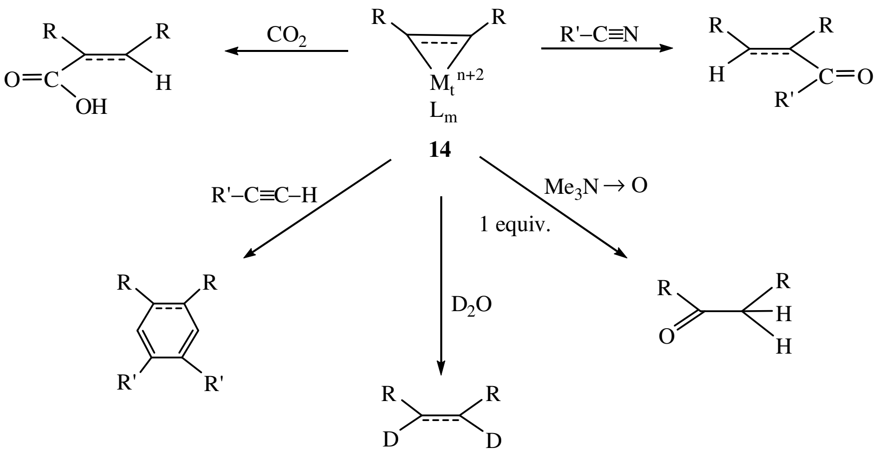
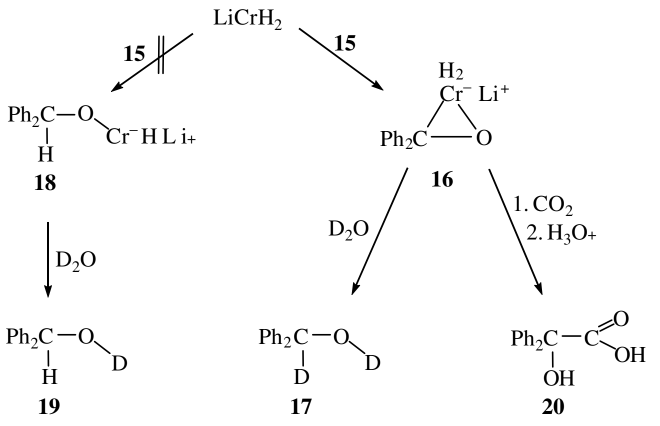
[1] This first hydrocarboxylation, via epichromation and subsequent carbonation, was achieved in 2000 by J.J. Eisch and F.A. Owuor, subsequent to the publication of the preparation and properties of $LiCrH_2$ in Ref. [15] and this work was cited in Ref. [5].



Scheme 2.



Scheme 3. Scheme 3 exemplifies such insertions with the generalized epimetallated product **14**, although in actuality a specific transformation may have been achieved via the epimetallation of any olefin or acetylene.



Scheme 4.

16 and not of hydrochromation to yield **18**. Were **18** to have been present, D_2O treatment would have yielded **19**, a product *not* formed (Scheme 4). Carbonation of **16** at low temperatures did provide benzilic acid (**20**), up to 65%, but always admixed with variable amounts of diphenylacetic acid, i.e., deoxygenated **20**.[1]

2. Results and discussion

Our recent synthesis and characterization of the analogous lithium vanadium(I) dihydride, $LiVH_2$ (**21**) as a biradical vanadium reagent[2] have shown it to have similar epimetallating action to $LiCrH_2$ but having much less the tendency to cleave C–O bonds.[3]

In the present study we have explored the utility of lithium vanadium(I) dihydride, $LiVH_2$ (**21**), for the hydrocarboxylation of carbonyl and imino derivatives in useful yields and with minimal formation of deoxyacids. Our model substrates for stepwise epivanadation and carbonation have been 9-fluorenone (**22**) and 9-fluorenone anil (**23**), as treated first with 2 equiv. of $LiVH_2$ (**21**) and then with CO_2, as shown in Scheme 5. Epivanadation of either **22**

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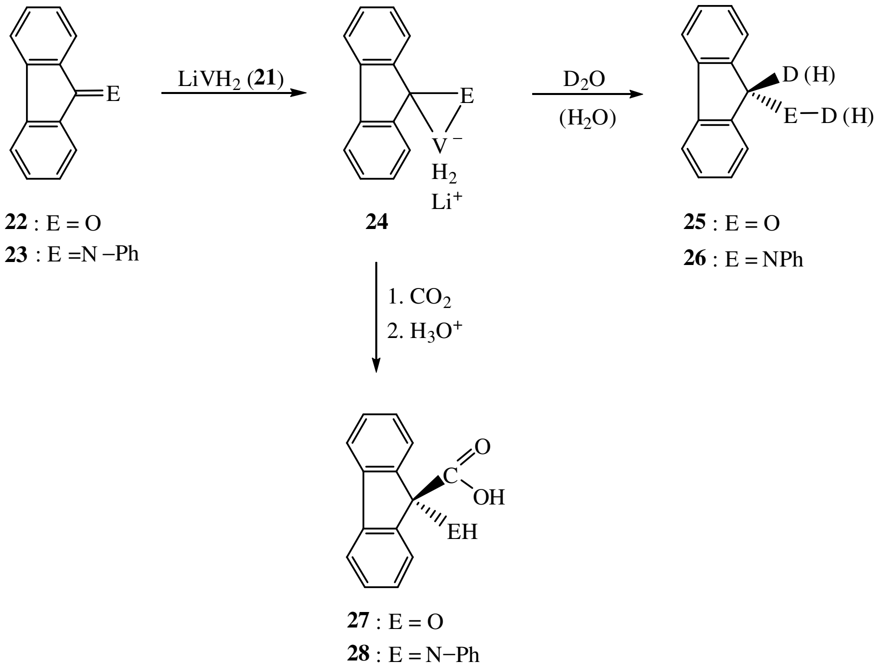
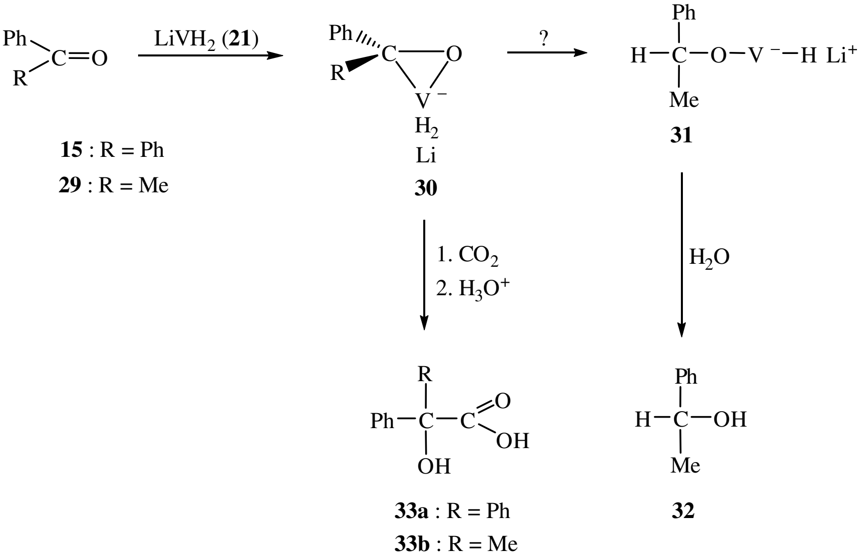
[2] The gasometric, infrared spectral and chemical characterization of **21** as having the empirical formula of $LiCrH_2$ is given in Ref. [13a]. The detailed EPR investigation of **21** establishing it as the lithium salt of the linear biradical VH_2 anion will also be described in the next publication in this series (Ref. [13a]).

[3] As a typical example, $LiCrH_2$ cleaves dibenzofuran and, at a slower rate, the solvent THF, so that *o*-phenylphenol and 1-butanol result upon hydrolysis. Under similar conditions, $LiVH_2$ cleaves neither ring.



Scheme 5.



Scheme 6.

or **23** and treatment of respective aliquots with D_2O showed that 9-fluorenol (**25**) and 9-(*N*-phenylamino)fluorene (**26**) had formed in yields of 84% and 95%, respectively, with the C_9 center deuteriated in over 90%. Thus, epimetallated intermediates of the type **24** had been successfully formed and maintained in the reaction mixtures. Subsequent carbonation and hydrolysis produced 75% of **27** and 65% of **28** in nonoptimized yields (Scheme 5).

Similar reactions between 2:1 molar mixtures of $LiVH_2$ (**21**) and either benzophenone (**15**) or acetophenone (**29**) gave up to 80% of the secondary alcohol, **32**, upon hydrolysis but separate treatment of such reaction mixtures with D_2O revealed that **32** was about 50:50 mixtures of PhRCD–OD and PhRCH–OH. That the crucial C–V bond in the epimetallated intermediate **30** was largely lost, possibly via **31**, was confirmed by carbonation. With benzophenone benzilic acid (**33a**) was formed in only 28% yield and 9% of diphenylacetic acid was produced as a by-product. With acetophenone the yield of 2-hydroxy-2-phenylpropionic acid (**33b**) was decreased to 22% (Scheme 6).

The failure of this epivanadation–carbonation sequence could be circumvented for benzophenone by using $LiV(CH_3)_2$ (**34**) in place of $LiVH_2$ (**21**). Having no H atoms directly on the vanadium center, the intermediate involved here, similar to **30,** should be less prone to the hydrogenolytic rearrangement of **30**–**31**. Indeed, this assumption appears to be the case: the yield of benzilic acid now rose to 65% with no contamination by any diphenylacetic acid. However, a similar treatment of acetophenone with **34** followed by carbonation did not increase the yield of **33b**.

Hence, we have now begun to extend our studies to the use of neutral metallating reagents, such as tributylvanadium, vanadium(I) chloride, titanium(II) isopropoxide and dibutyltitanium diisopropoxide. We have thereby found that titanium(II) isopropoxide can epititanate benzophenone quantitatively to give, upon hydrolysis, benzhydrol, without producing any benzopinacol, the reductive dimer [6]. This epimetallated product has now been shown to undergo carbonation to yield benzilic acid cleanly and exclusively in 40% yield. By employing other neutral vanadium or titanium reagents for the epimetallation of benzophenone, as well as for other carbonyl and imino derivatives, we aspire to arrive at a more general process for producing 2-hydroxy- or 2-amino acids from ketones or imines.

3. Conclusion

We have demonstrated the feasibility of achieving the hydrocarboxylation of a number of sterically substituted ketones and an imine to give useful yields of 2-hydroxy and 2-amino acids, respectively, by the two-step process of epivanadating the C═E linkage (E = O, NR) with $LiVH_2$ or $LiV(CH_3)_2$ and then carbonating the vanadaoxa- or vanadaazacyclopropane intermediate. Further studies using vanadium(I) or titanium(II) complexes as the direct epimetallating agent or dibutyltitanium diisopropoxide as the transfer-epimetallating agent offer the possibility of extending the applicability of such a hydrocarboxylation process.

4. Experimental

4.1. Instrumentation, analysis and starting reagents

All reactions were carried out under a positive pressure of anhydrous, oxygen-free argon. All solvents employed with organometallic compounds were dried and distilled from a sodium metal–benzophenone ketyl mixture prior to use [17]. The IR spectra were recorded with a Perkin-Elmer instrument, model 457 and samples were measured either as mineral oil mulls or as KBr films. The NMR spectra (^1H and ^{13}C) were recorded with a Bruker spectrometer, model EM-360, and tetramethylsilane (Me_4Si) was used as the internal standard. The chemical shifts reported are expressed in the δ-scale and in parts per million (ppm) from the reference Me_4Si signal. The GC/MS measurements and analyses were performed with a Hewlett–Packard GC 5890/Hewlett–Packard 5970 mass-selective detector instrument. The gas chromatographic analyses were carried out with a Hewlett–Packard instrument, model 5880, provided with a 6 ft. OV-101 packed column or with a Hewlett–Packard instrument, model 4890, having a 30 m SE-30 capillary column, respectively. Melting points were determined on a Thomas–Hoover Unimelt capillary melting point apparatus and are uncorrected.

The VCl_3, VCl_4, methyllithium in ethyl ether and *n*-butyllithium in hexane were obtained from commercial sources, as were 9-fluorenone and benzophenone, in at least 97% purity. The 9-fluorenone anil, m.p. 88 °C, was prepared from 9-fluorenone and aniline by a published method [18].

*4.2. Preparation of lithium vanadium(I) dihydride (**21**) from vanadium(IV) chloride*

Although $LiVH_2$ (**21**) can be prepared from the interaction of either VCl_3 or VCl_4 with appropriate equivalents of *n*-butyllithium in THF and the resulting reagent **21** in solution has very similar properties, the reagent **21** resulting from VCl_4 is more stable upon solvent removal: samples of **21** isolated by the VCl_4-route consistently give 2.5 ± 0.1 equiv. of H_2 at STP by gasometric analysis with glacial acetic acid, while solvent-free samples of **21** isolated from the VCl_3-method give considerably less hydrogen upon such analysis (<2 equiv. H_2). Therefore, the preparation of **21** by the VCl_4-route is described here.

To a solution of VCl_4 (920 mg, 4.8 mmol) in 30 mL of THF at −78 °C was slowly added *n*-BuLi (14.9 mL, 23.8 mmol., 1.6 M in hexane) and the resulting mixture allowed to stir for 30 min. The solution was then rapidly brought to RT and stirred for 2 h, during which time the color changed from reddish brown to black. A solution of the desired carbonyl or imino substrate in THF was then introduced.

*4.3. Preparation of lithium dimethylvanadate(I) (**34**)*

Anhydrous VCl_3 (750 mg, 4.8 mmol) in 30 mL of THF at −78 °C was admixed with methyllithium (11.9 mL, 19.1 mmol, 1.6 M in ethyl ether) and the mixture then stirred for 30 min. After being brought to RT and then stirred for 2 h, the mixture turned from purple to black. Appropriate gasometric analyses of reagent **34** have yet to be performed and its suggested structure as lithium dimethylvanadate(I) (**34**) is based upon the analogous reaction of VCl_3 with 4 equiv. of *n*-BuLi, where successively $LiV(n\text{-Bu})_4$, then $LiV(n\text{-Bu})_2$ and finally $LiVH_2$ would be formed. With the methylated analog, $LiV(CH_3)_2$ would be the final product, since this intermediate cannot undergo β-hydride elimination. Also when admixed with ketones, **34** epimetallates the C═O group and does not insert the C═O group into a V–CH_3 bond.

4.4. Preparation of titanium(II) isopropoxide

To a solution of titanium(IV) isopropoxide (5.95 mL, 20 mmol) dissolved in anhydrous, deoxygenated THF (30 mL) at −78 °C was added slowly *n*-butyllithium

(25 mL in hexane, 40 mmol) whereupon the solution turned light brown and then brown. The reaction mixture was stirred overnight (18 h) as the reaction temperature was allowed to rise to room temperature. At this point, a black solution of titanium(II) isopropoxide (**22**) was obtained [6].

4.5. Reaction of lithium vanadium dihydride (**21**) with 9-fluorenone (**22**)

4.5.1. Workup with D_2O

When a 1:2 mole ratio of **22** and **21** was employed at RT, the reaction led to the disappearance of any coupled products and an increase in **25**. Thus a solution of $LiVH_2$ (**21**) (3.9 mmol) in 30 mL THF was allowed to react with a solution of 9-fluorenone (**22**) (350 mg, 1.9 mmol) in 10 mL THF and allowed to stir for 12 h from −78 °C to room temperature. Hydrolytic workup of the reaction mixture yielded 690 mg of a pale yellow solid. The crude product was then washed with pentane to give 30 mg of 12% of fluorene and 4% of unreacted 9-fluorenone, while the pentane-insoluble residue was found to be 9-fluorenol **25** (84% yield).

In a separate experiment designed to establish the source of the benzhydryl C_9–H in **25** a solution of $LiVH_2$ (4.8 mmol) (**21**) in 40 mL was again allowed to react with 9-fluorenone (**22)** (440 mg, 2.4 mmol) in 10 mL THF at room temperature for 12 h. Workup of both halves of the reaction mixture, using ordinary water and deuterium oxide, respectively, gave pale yellow solids. The ^{1}H NMR spectrum of the reaction solution worked up with D_2O showed the presence of 82% 9,*O*-dideuterio-9-fluorenol (**25**).

4.5.2. Workup with carbonation

The presence of a carbon–vanadium bond in the oxavanadacyclopropane intermediate, established by incorporation of deuterium at the C_9 carbon in **25**, encouraged the investigation of the reactivity of such bonds to carbon dioxide. Thus a solution of $LiVH_2$ (**21**) (8.9 mmol) in 120 mL THF was allowed to react with a solution of 9-fluorenone (**22**) (4.5 mmol, 810 mg) in 20 mL THF room temperature for 12 h. The resulting reaction mixture was then carbonated using a stream of dry carbon dioxide at −78 °C and left to warm up to room temperature for 7 h to give 810 mg of a pale yellow solid after hydrolytic workup and isolation from the aqueous layer. The ^{1}H and ^{13}C NMR spectra recorded in DMSO-d_6 showed the presence of 75% 9-hydroxy-9-fluorenecarboxylic acid (**27**) and 6% of 9-fluorenecarboxylic acid; these products were identified by NMR spectral comparison with spectra of authentic samples.

*9-Hydroxy-9-fluorenecarboxylic acid (**27**)*

^{1}H NMR: 7.1–7.6, (m, 4H); 7.8 (d, 2H).
^{13}C NMR: 82.27, 120.09, 123.94, 125.66, 127.83, 140.26, 146.95, 173.91.

9-Fluorenecarboxylic acid

^{1}H NMR: 7.55 (d, 2H), 7.65 (d, 2H), 7.40–7.20 (m, 6H), 4.85 (s, 1H) ppm.
^{13}C NMR: 171.6, 141.1, 140.7, 127.7, 127.1, 125.5, 119.9, 53.1 ppm.

4.6. Reaction of lithium vanadium dihydride (**21**) with 9-fluorenone anil (**23**)

4.6.1. Workup with D_2O

The deuteriolytic workup of a 1:2 mixture of **23** and **21** in THF, conducted analogously with the corresponding 9-fluorenone experiment (Section 4.5) showed that the 9-(*N*-phenylamino)fluorene was 95% dideuteriated in the form of **26**.

4.6.2. Workup with carbonation

A solution of 9-fluorenoneanil (**23**) (80 mg, 0.5 mmol) in 5 mL tetrahydrofuran was allowed to react with a solution of $LiVH_2$ (**21**) (1.0 mmol) in 5 mL tetrahydrofuran at room temperature for 12 h. The resulting mixture was then carbonated with dry carbon dioxide at −78 °C and gradually brought to room temperature over 12 h to give after hydrolytic workup 65% of 9-(*N*-phenylamino)-9-fluorenecarboxylic acid (**28**). Structural elucidation of **28** was achieved by ^{1}H, ^{13}C and, decisively, by electron-spray mass spectrometry, in order to observe the parent ion peak of 324 ($301 + Na^+$ complex).

^{1}H ($CDCl_3$) δ: 7.81–7.98(d, 2H), 7.46–7.23(m, 8H), 6.83(t, 2H), 6.52(t, 1H), 6.07(d, 1H) ppm.
^{13}C ($CDCl_3$) δ: 176.7, 143.6, 141.1, 134.7, 129.4, 128.6, 128.1, 123.7, 120.6, 118.1, 114.6, 70.9 ppm. MS (electrospray with NaI, *m*/*e*): 165.1, 181.1, 109.0, 210.1, 256.1, 302.1, 303.1, 324.1 ($M^+ + 23$ of Na^+).

4.7. Epivanadations of benzophenone and acetophenone with lithium vanadium(I) dihydride or lithium dimethylvanadate(I)

These epivanadations and their workup by carbonation were conducted analogous to the foregoing procedures for 9-fluorenone and 9-fluorenone anil. The benzilic acid and 2-hydroxy-2-phenylpropionic acid products were identified by NMR spectral comparison with spectra of authentic samples.

4.8. Epititanation of benzophenone with titanium(II) isopropoxide (**22**) and subsequent carbonation

A solution of 20 mmol of titanium(II) isopropoxide generated in 60 mL of THF–hexane (Section 4.4) and 10 mmol of benzophenone (**15**) (10 mmol) was cooled to −78 °C and treated with a steady stream of dry CO_2 gas as the temperature of the reaction mixture was raised to RT over 3 h. Thereafter, the reaction mixture was carbonated continuously as the solution was maintained at reflux for 3 h. Hydrolytic workup with aqueous N–HCl yielded an organic extract. This solution was extracted with aqueous NaOH. The NaOH aqueous extract was acidified with aqueous N–HCl to precipitate the pure benzilic acid, m.p. 148–150 °C. Two identical runs of this reaction gave yields of 39% and 40% of this acid.

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