TLC, Merck aluminum sheets precoated with Kieselgel 60 F_{254} or Aluminum oxide F_{254} were used. For other general experimental information see ref 7. Physical and spectroscopic data of products are given in Table III.

General Procedure for the Lithiation of 1a-5a and Reaction with Electrophiles. Preparation of 1-6c-f and 7-9. A solution of the lithiating agent (5 mmol) in THF (5 mL) at -80 °C was treated dropwise with a solution of **1a-5a** (5 mmol) in THF (5 mL). The reaction mixture was stirred at -80 °C for 30 min, and then the electrophile (5 mmol) was added neat. The mixture was stirred for an additional 30 min then hydrolyzed by the addition of saturated aqueous NH_4Cl solution at -80 °C and extracted in ether.

General Procedure for the Oxidation of Alcohols 2c-5c. Preparation of 2g-5g. A solution of the alcohol **2c-5c** (2 mmol) in anhydrous acetone (6 mL) was treated portionwise with powdered CrO_3 (600 mg, 6 mmol) at 0 °C with vigorous stirring. The mixture was stirred for 10 min at rt, and then the excess oxidizing agent was destroyed by the addition of 2-propanol (1 mL), and stirring was continued for 10 min. The mixture was poured into saturated aqueous $NaHCO_3$ solution (30 mL). The solids were filtered off and washed with acetone (2 mL) and ether (50 mL). The product was extracted from the filtrates in ether.

4-Hydrazino-3-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine (10a). A suspension of **1g** (253 mg, 1 mmol) in EtOH (3 mL) was treated with anhydrous hydrazine (0.08 mL, 2.5 mmol) with stirring. The solution was heated to reflux, filtered while hot, and concentrated. The product, which crystallized out, was filtered and washed with EtOH and ether; yield 84%.

1-Methyl-4-(1-methylhydrazino)-3-phenyl-1*H*-pyrazolo-[3,4-*d*]pyrimidine (10b). A suspension of **1g** (253 mg, 1 mmol) in MeOH (2 mL) was treated with methylhydrazine (0.26 mL, 5 mmol) with stirring. The mixture was stirred for 2 h at rt, the solvents were removed under reduced pressure, and the product was taken in ether; yield 80%.

5-Benzoyl-6-chloro-4-(phenylamino)pyrimidine. A solution of **1g** (506 mg, 2 mmol), aniline (0.22 mL, 2.4 mmol), and Et_3N (1 mL) in benzene (10 mL) was heated under reflux for 12 h and then left 16 h at rt. The solvents were removed under reduced pressure, and the product was taken in CH_2Cl_2. The solution was washed with water and dried (Na_2SO_4). The product was further purified by chromatography; yield 92%, mp 125-126 °C (CH_2Cl_2/hexane): ^1H NMR ($CDCl_3$) δ 7.14 (t, 1 H, J = 2.0 Hz), 7.33 (t, 2 H, J = 2.0 Hz), 7.44-7.56 (m, 4 H), 7.62 (t, 1 H, J = 2.0 Hz), 7.82 (d, 2 H, J = 2.0 Hz), 8.39 (s, 1 H), 8.52 (s, 1 H); ^{13}C NMR ($CDCl_3$) δ 112.88 (s), 122.08 (d), 124.96 (d), 128.81 (d), 129.20 (d), 134.14 (d), 137.17 (s), 157.96 (s), 158.37 (d), 159.19 (s), 194.63 (s). Anal. Calcd for $C_{17}H_{12}ClN_3O$: C, 65.92; H, 3.91; N, 13.57. Found: C, 66.28; H, 4.15; N, 13.66.

5-Phenylpyrimido[4,5-*b*]quinolin-4(3*H*)-one (11). A homogenized mixture of the previous product (310 mg, 1 mmol) and PPA (3.0 g) was heated at 100 °C bath temperature for 10 min. On cooling the mixture was hydrolyzed with water and neutralized with saturated aqueous K_2CO_3 solution. The product, which crystallized out, was filtered and washed with water and MeOH; yield 97%.

4-Chloro-5-phenyl-11*H*-pyrimido[4,5-*b*][1,5]benzodiazepine (12). A suspension of 1,2-phenylenediamine (130 mg, 1.2 mmol) in anhyd benzene (4 mL) was treated with Et_3N (0.14 mL, 1 mmol), and the mixture was stirred at 45 °C until a clear solution was formed. Then, a solution of **1g** (253 mg, 1 mmol) in benzene (3 mL) was added dropwise with stirring and the reaction mixture was refluxed for 1 h. Solvents were removed under reduced pressure and the product was purified by chromatography on silica gel using benzene/ether as the eluent; yield 87%.

Registry No. **1a**, 3934-20-1; **1c**, 130825-16-0; **1d**, 62803-30-9; **2a**, 1193-21-1; **2c**, 109574-98-3; **2d**, 134031-19-9; **2g**, 109575-04-4; **3a**, 3764-01-0; **3c**, 134031-16-6; **3d**, 134031-20-2; **3g**, 91546-44-0; **4a**, 26452-80-2; **4c**, 134031-17-7; **4d**, 134031-21-3; **4f**, 134031-24-6; **4g**, 134031-25-7; **5a**, 2402-78-0; **5c**, 58584-77-3; **5d**, 134031-22-4; **5e**, 58584-94-4; **5f**, 55304-73-9; **5g**, 118067-08-6; **6c**, 134031-18-8; **6d**, 134031-23-5; **6e**, 39621-00-6; **6f**, 113293-70-2; **7**, 54503-93-4; **8**, 134031-26-8; **9**, 134031-27-9; **10a**, 134031-28-0; **10b**, 134031-29-1; **11**, 31407-32-6; **12**, 134031-30-4; PhCHO, 100-52-7; Me_3SiC, 75-77-4; MeI, 74-88-4; *N*-formylpiperidine, 2591-86-8; hydrazine, 302-01-2; methylhydrazine, 60-34-4; aniline, 62-53-3; 1,2-phenylenediamine, 95-54-5; 5-benzoyl-6-chloro-4-(phenylamino)pyrimidine, 134031-31-5.

Supplementary Material Available: Regioselectivity of lithiation of **5a** as determined by reference addition to PhCHO and characterization data for previously reported compounds (2 pages). Ordering information is given on any current masthead page.

Silica Gel Supported Zinc Borohydride. 2.[1] Regioselective 1,2-Reduction of Conjugated Ketones and Aldehydes to the Corresponding Allylic Alcohols

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The supported-reagent technique has attracted considerable interest among synthetic chemists in recent years.[2] Advantages frequently claimed in favor of supported reagents compared with their homogeneous counterparts are increased yields, mild conditions, and clean and rapid reactions, but examples of regio-, stereo-, and chemoselective control using supported reagents are rather few.[3] We have recently introduced silica gel supported zinc borohydride for the regio- and stereoselective reductive cleavage of unsymmetrical epoxides[1] and herein wish to disclose another application of this reagent for regioselective 1,2-reduction of conjugated ketones and aldehydes.

Selective reduction of conjugated ketones and aldehydes to the corresponding allylic alcohols is a challenging problem, since it is usually associated with varying amounts of concomitant reduction of the double bonds. This leads to saturated alcohols and/or ketones, due to competing 1,2- vs 1,4-attack by hydride.[4] Considerable progress has been made in the development of various reducing agents for this purpose,[5] but few have proven very

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Table I. Reduction of Conjugated Carbonyl Compounds to the Corresponding Allylic Alcohols with Silica Gel Supported Zinc Borohydride

entry	starting carbonyl compd	time (h)	yield[a] (%)
1	3-methylcyclohex-2-en-1-one	8	80
2	4-carbethoxy-3-methylcyclohex-2-en-1-one[5j]	8	82[b]
3	(*S*)-(+)-carvone[5l]	8	80[c]
4	3-carbomethoxycyclohex-2-en-1-one	8	85
5	α-ionone[5l]	8	85
6	cyclopent-2-en-1-one	8	20 (50)[d]
7	cyclohex-2-en-1-one	8	15 (55)[d]
8	$PhCH{=}CHCOCH_3$	7	85
9	mesityl oxide	8	65
10	cinnamaldehyde	7	87
11	citral	7	88
12	crotonaldehyde	7	80
13	4-nitrobenzaldehyde	7	85

[a] Yield of isolated pure products. [b] As mixture of isomers (70:30) by GLC. [c] As mixture of isomers (60:40) by GLC. [d] Yields in parentheses refer to those obtained by carrying out the reaction at −23 °C (entry 6) and −78 °C (entry 7) for 8 h without any solvent.

Table II

carbonyl compd	% composition of 1,2- vs 1,4-product (GC)	% yield of isolated allylic alcohol
3-methylcyclohex-2-en-1-one	50:50	38
(*S*)-(+)-carvone	60:40	45
$PhCH{=}CHCOCH_3$	50:50	40
citral	55:45	40
cyclopent-2-en-1-one	15:85	
cyclohex-2-en-1-one	10:90	

general and practical in scope. Thus, there is a need for new reducing agents that are reliable, easy to use, and inexpensive. We have found that our recently developed silica gel supported zinc borohydride[1] provides an efficient and highly selective reduction of conjugated ketones and aldehydes to the corresponding allylic alcohols.

In a typical procedure, the conjugated carbonyl compound was treated with silica gel supported zinc borohydride in tetrahydrofuran at −5 to −10 °C for 7–8 h and the corresponding allylic alcohol was isolated in a pure state by hydrolysis and simple ether extraction. The results are reported in Table I.

As shown in Table I, structurally varied conjugated ketones and aldehydes were reduced to the corresponding allylic alcohols in good yields. No appreciable 1,4-reduction products were observed or isolated except in the cases of cyclopentenone and cyclohexenone. But, much improvement was observed when reduction was carried out with these two ketones in the absence of solvent (entries 6, 7). Several easily reducible functional groups, such as carboxylic ester, double bond, and nitro, remained unaffected under these conditions.

Zinc borohydride in DME has been reported[6] to effect reduction of conjugated ketones to the allylic alcohols in the synthesis of prostaglandins, but we found this combination to produce substantial amounts of 1,4-addition products in simple cyclic and acyclic carbonyl compounds.[7] Our supported reagent eliminates this drawback. Moreover, this reagent has unique and uniform reducing properties toward both cyclic and acyclic enones different from those obtained with sodium cyanoborohydride[5l] and diisobutylaluminum hydride.[5n] Compatibility with a variety of normally reducible functional groups makes this procedure more attractive and useful for selective reductions in multifunctional molecules. Thus, this reagent avoids the disadvantages of strong reducing agents like DIBALH.[5n] Though reductions of cyclopentenone and cyclohexenone with this supported reagent are not as satisfactory as those obtained with $NaBH_4/CeCl_3$,[5h] DIBALH,[5n] and 9-BBN,[5j] significant improvement during neat reduction is interesting[8] compared with the results in solution.[7]

In conclusion, silica gel supported zinc borohydride thus appears to be a mild and efficient reagent for the reduction of a variety of conjugated aldehydes and ketones to the corresponding allylic alcohols in high yields. Moreover, the easy availability of this reagent makes this simple procedure extremely attractive and a practical alternative to the existing methods,[5] and we believe this will find general acceptance in organic synthesis.

Experimental Section

^1H NMR spectra were recorded at 60 or 200 MHz in CCl_4 and $CDCl_3$ solutions. GLC was done using a SE-30 column (2 m) and N_2 as carrier gas. Thin-layer chromatography was done on precoated silica gel plates (Eastman Kodak Co.). $Zn(BH_4)_2$ in 1,2-dimethoxyethane (DME) was prepared from $ZnCl_2$ and $NaBH_4$ according to the reported procedure.[6b] THF was distilled from Ph_2CO–K under N_2 immediately prior to use.

Preparation of Silica Gel Supported Zinc Borohydride. A solution of $Zn(BH_4)_2$ (285 mg, 3 mmol) in DME (3 mL) was added to silica gel HF 254 (1 g) and stirred at room temperature for 30 min. Solvent was then evaporated under reduced pressure at room temperature to give the supported reagent, which was used for the reduction of conjugated carbonyl compound (1 mmol) on the same or next day.

General Procedure for Reduction. 3-Methylcyclohex-2-en-1-one (110 mg, 1 mmol) was stirred with the silica gel supported $Zn(BH_4)_2$, prepared as in the previous text, in THF (5 mL) at −5 to −10 °C (ice–salt bath) under N_2. Stirring was continued for 8 h until completion of the reaction as monitored by TLC. The reaction mixture was then decomposed by careful dropwise addition of water and filtered through Celite. The filtrate was extracted with Et_2O (3 × 10 mL), and the extract was washed with saturated aqueous solution of $NaHCO_3$ and brine. It was then dried (Na_2SO_4) and evaporated to leave the product. The crude product was chromatographed through a short column of silica gel to leave a pure alcohol (90 mg, 80%).

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(8) Reduction in the absence of solvent is now being explored for further useful applications.

This procedure was followed for the reduction of the other conjugated carbonyl compounds. The allylic alcohols obtained were purified (≥95%) by column chromatography (distillation was avoided to prevent loss of material due to decomposition or polymerization). The products were identified and the purities were checked by comparison with authentic samples (TLC, IR, and ^{1}H NMR).

Spectral data for 3-carbomethoxycyclohex-2-en-1-ol (entry 4): IR (neat) 3200–3500 (broad), 1715 cm^{-1}; ^{1}H NMR (CCl_4) δ 1.2–2.3 (m, 6 H), 3.62 (s, 3 H), 3.84 (broad s, 1 H), 4.20 (broad, 1 H), 6.81 (m, 1 H) (homogeneous by GLC and TLC).

Reduction of 3-Methylcyclohex-2-en-1-one on a Multigram Scale. 3-Methylcyclohexenone (5.5 g, 0.05 mol) was added dropwise to a stirred suspension of $Zn(BH_4)_2$ (14.25 g, 0.15 mol) supported on silica gel (30 g; (prepared as in the previous text from the solution (140 mL) of $Zn(BH_4)_2$ in DME and silica gel) in THF (100 mL) under N_2 and stirred further for 8 h at −5 to −10 °C. The reaction was worked up as in the previous text and the crude product was chromatographed over silica gel to leave the pure alcohol (4.56 g, 80%).

Alternatively, supported reagent can be added in portions to the solution of 3-methylcyclohexenone in THF to get same result.

Reduction of Cyclohex-2-en-1-one with the Supported Reagent without Any Solvent. Neat cyclohexenone (96 mg, 1 mmol) was added to the solid supported reagent, prepared as in the previous text, at −78 °C under N_2, and stirring was continued for 8 h (monitored by TLC). The reaction mixture was decomposed with careful dropwise addition of H_2O and worked up as in the previous text to furnish pure cyclohex-2-en-1-ol (54 mg, 55%) after chromatography. The same procedure was followed for the reduction of cyclopentenone at −23 °C.

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Four-Directional Building Blocks for the Synthesis of Cascade Polymers[1]

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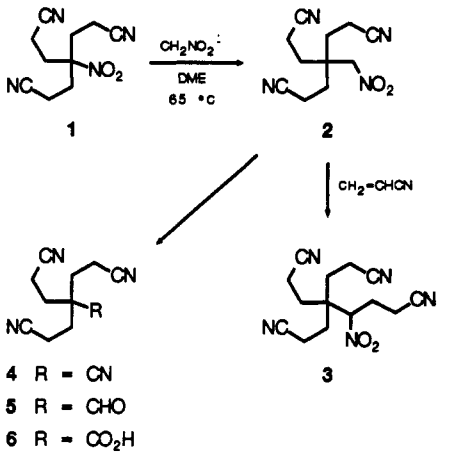
Tris(β-cyanoethyl)nitromethane (1) has become a convenient and inexpensive starting material for the construction of cascade polymers ("arborols")[2] and Micellanes.[3] Although transformations have been reported involving the nitrile functionality of 1, little attention has been given to the conversion of the nitro group into carboxylic acid derivatives.[4] Since nitro groups attached to tertiary carbon atoms can undergo radical anionic displacement reactions,[5] we decided to investigate the potential use of 1 as a source of new building blocks for cascade polymers.

Scheme I



Homologation was easily accomplished by treatment of the sodium salt of nitromethane with 1, thus providing a quaternary C-center having a $-CH_2NO_2$ substituent. Subsequent transformations of the $-CH_2NO_2$ moiety (Scheme I) gave rise to novel polyfunctionalized neopentane cores.

Although the reaction of 4 mol of nitromethyl anion with 1 mol of 1 in DMSO in the presence of an external light source did not occur at 25 °C,[5,6] reaction commenced spontaneously at 60–65 °C and was terminated after ~30 min, providing 2 in 64% yield. Extended reaction times caused diminished yields of isolated product. Compound 2 was easily characterized by the appearance of a new peak at δ 78.2 in the ^{13}C NMR spectrum for the CH_2NO_2 and a notable shift (Δδ 50) for the quaternary carbon. Interestingly, as noted earlier,[5] no reaction was observed when DMF was used as a solvent. Further substitution of 2 with acrylonitrile in the presence of Triton-B or DBU was restricted to the introduction of only one β-cyanoethyl group, affording 3 in 40% yield. The ^{13}C NMR spectrum of 3 shows a peak at δ 92.2 ($CHNO_2$) and other peaks for the unique cyanoethyl moiety. Presumably, steric factors hamper any further approach of a second acrylonitrile molecule. It should be noted that the generally facile replacement of the nitro group in 1 by a β-cyanoethyl moiety by using tri-*n*-butyltin hydride[7] failed completely.

Transformations of a primary nitro group into other functionalities have been reported.[8] Conversion of the $-CH_2NO_2$ moiety into nitrile 4 by reaction with PCl_3 in pyridine was straightforward.[9] The appearance of two C≡N peaks in the ^{13}C NMR spectrum at δ 119.6 and 120.3 in an approximate 1:3 ratio confirmed the structure of 4. Aldehyde 5 was prepared in excellent yield by the oxidation of 2 with $KMnO_4$ in aqueous potassium tetraborate solution.[10,11] The structural assignment is supported by the new peak in the ^{13}C NMR spectrum at δ 204.6 for the

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