TLC analysis of the reaction mixture showed a mixture of products.

A reference sample of (*S*)-1 was synthesized using Schollkopf's method, and this sample (90–92% ee) had $[\alpha]_D = +25.32°$ ($c = 0.97$, H_2O).[12] The sample of (*S*)-1 obtained by the method described in this paper had $[\alpha]_D = +27.30°$ ($c = 1$, H_2O). This result provided unequivocal evidence that the allylation of **2b** proceeded with retention of configuration. Our sample of 1 was >98% ee as determined by thin-layer chromatography on a commercially available Chiralplate.[13,14] This value was independently verified using the NMR method of Kellogg et al.[5f]

The method described in this paper provided α-alkyl α-amino acids in good overall yield and with high optical purity. The mild conditions required for the final deblocking of alkylated intermediates made this a useful method for the synthesis of amino acids containing acid sensitive side chains.

Acknowledgment. We wish to thank Drs. Merrill Nuss and Andy Zetzker, Ms. Ruth Stanaszek, and Mr. David Whittern for performing numerous NMR experiments.

Supplementary Material Available: All experimental procedures and X-ray data for **2b** (22 pages); structure factors for **2b** (6 pages). Ordering information is given on any current masthead page.

(12) The sample of (*S*)-1 prepared in ref 4 was characterized as the *N*-acetyl derivative. We repeated this synthesis and obtained the free amino acid which was used as a reference standard.

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(14) Optical purity values (% ee) were calculated on the basis of specific rotation values in the literature and/or using the NMR method described in ref 5f. The optical purity of α-allylphenylalanine was determined by TLC analysis on a commercially available Chiralplate using a racemic sample of 1, which was prepared from the formaldehyde oxazolidinone. Optical rotation values for the amino acids synthesized in this work are reported in the supplementary material.

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Diels–Alder Reactions of 1-Aza-1,3-butadienes: Room Temperature, Endo-Selective $LUMO_{diene}$-Controlled [4 + 2] Cycloaddition Reactions of *N*-Sulfonyl-4-(ethoxycarbonyl)-1-aza-1,3-butadienes

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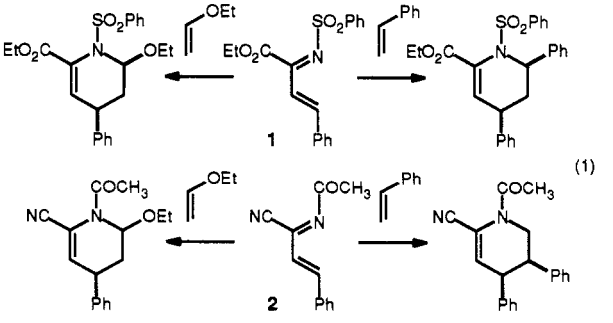
Summary: The room temperature, endo-selective $LUMO_{diene}$-controlled Diels–Alder reactions of *N*-(phenylsulfonyl)- and *N*-(methylsulfonyl)-4-(ethoxycarbonyl)-1-aza-1,3-butadiene (**3–4**) are described, and the results represent a demonstration of the [4 + 2] cycloaddition rate acceleration achievable through noncomplementary azadiene substitution.

The Diels–Alder 4π participation of simple 1-aza-1,3-butadienes, α,β-unsaturated imines, is rarely observed and typically suffers low conversions, competitive imine addition, and/or imine tautomerization precluding productive [4 + 2] cycloaddition.[1,2] However, in recent efforts we have demonstrated the general 4π participation of stable *N*-(phenylsulfonyl)-1-aza-1,3-butadienes in regiospecific and endo-specific inverse electron demand Diels–Alder reactions suitable for the diastereoselective preparation of 1,2,3,4-tetrahydropyridines and that the complementary substitution of the electron-deficient dienes with a C-3 electron-withdrawing substituent predictably accelerates their rate of participation in a $LUMO_{diene}$-controlled [4 + 2] cycloaddition reaction.[3] Extensions of these studies have illustrated that the noncomplementary C-2 addition of an electron-withdrawing substituent (CO_2Et) to the *N*-sulfonyl-1-aza-1,3-butadiene predictably accelerates the diene participation in $LUMO_{diene}$-controlled [4 + 2] cycloaddition reactions, maintains the expected cycloaddition regioselectivity, and maintains or enhances the endo diastereoselectivity (≥20:1), and that the reactions display characteristics consistent with a concerted $LUMO_{diene}$-controlled Diels–Alder reaction.[4]

Concurrent with these efforts, Fowler and Teng[5] have examined the intra- and intermolecular [4 + 2] cycloaddition reactions of *N*-acyl-2-cyano-1-aza-1,3-butadienes and have disclosed that such dienes participate in [4 + 2] cycloaddition reactions with electron-rich dienophiles with a reactivity, regioselectivity, and diastereoselectivity comparable to the *N*-sulfonyl-2-(ethoxycarbonyl)-1-aza-1,3-butadienes. However, in contrast to our disclosure of the clean observation of the 2-aryl-1,2,3,4-tetrahydropyridine cycloaddition regioisomer derived from the [4 + 2] cycloaddition of styrenes with **1**, Fowler and Teng have described the observation of mixtures of 3-aryl- and 2-aryl-1,2,3,4-tetrahydropyridines (8–1:1, respectively) with **2** (eq 1). Consequently, in efforts to define the origin of



(1)

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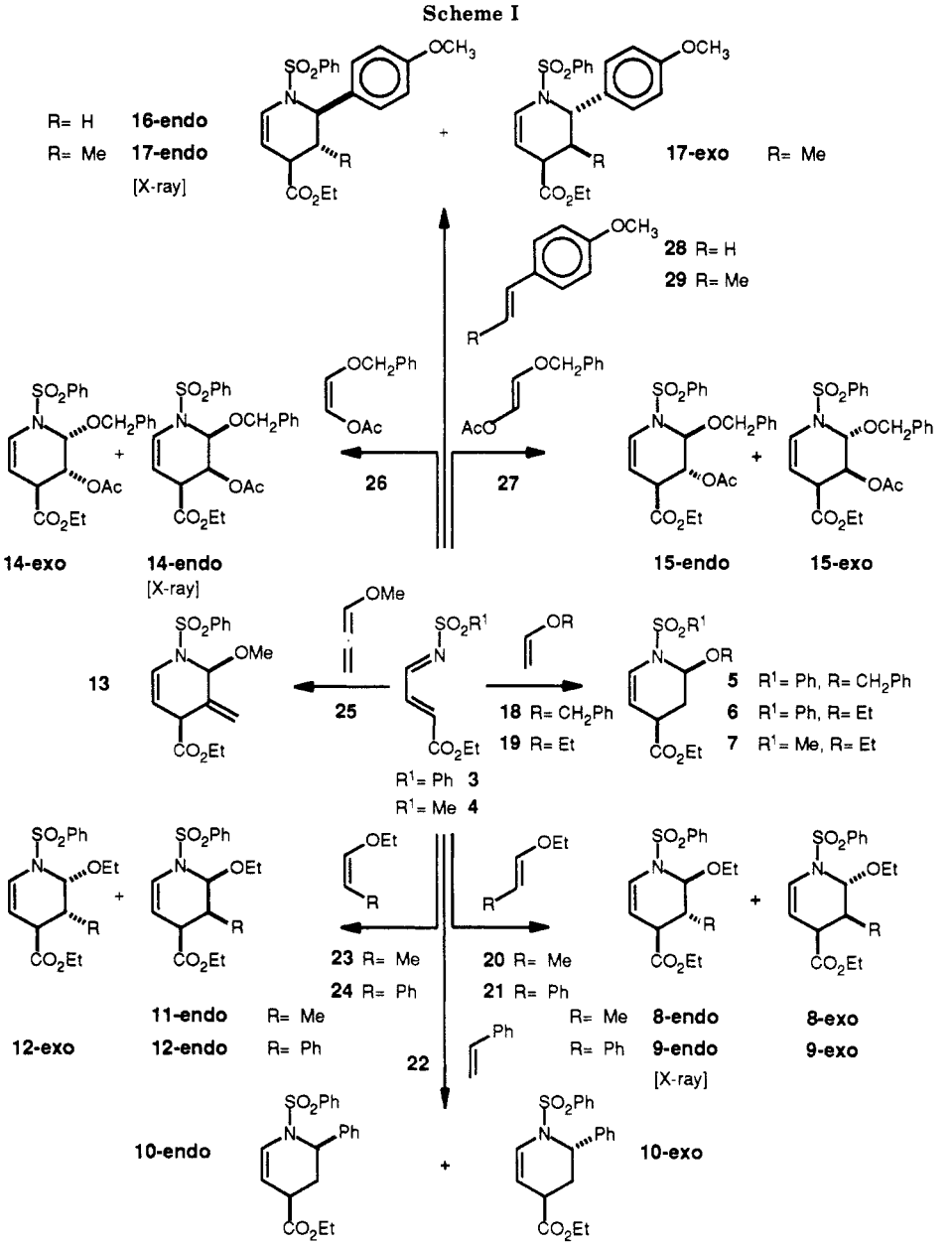
Scheme I



the regioselectivity differences observed in the two systems, we have examined the [4 + 2] cycloaddition reactions of *N*-(phenylsulfonyl)- and *N*-(methylsulfonyl)-4-(ethoxycarbonyl)-1-aza-1,3-butadiene (**3–4**) which further demonstrates that the *non*complementary C-4 addition of an electron-withdrawing group (CO_2Et) to the electron-deficient 1-aza dienes accelerates their 4π participation in $LUMO_{diene}$-controlled [4 + 2] cycloaddition reactions and maintains the [4 + 2] cycloaddition regioselectivity and endo diastereoselectivity of the parent *N*-sulfonyl aza dienes[3,4] and that the [4 + 2] cycloaddition reactions display characteristics consistent with concerted $LUMO_{diene}$-controlled [4 + 2] cycloaddition reactions.

Controlled ozonolysis of ethyl sorbate[6] followed by condensation of ethyl 4-oxo-2-butenoate with benzene- or methanesulfonamide (0.5 equiv of $TiCl_4$, CH_2Cl_2, 0 °C for 8 h) provided *N*-(phenylsulfonyl)- and *N*-(methylsulfonyl)-4-(ethoxycarbonyl)-1-aza-1,3-butadiene (**3–4**, 60–46%).[3,7-9] The results of a survey of [4 + 2] cycloaddition reactions of **3–4** with a full range of dienophiles are summarized in Scheme I and Table I. The structure and stereochemistry of the [4 + 2] cycloadducts were assigned initially based on diagnostic ^1H NMR chemical shifts and coupling constants,[10] were established firmly through NOE difference experiments,[10b] and were unam-

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(9) All new products exhibit ^1H NMR, ^{13}C NMR, IR, MS, and HRMS or CHN analyses consistent with the assigned structure. All yields of cycloadducts are based on pure material isolated by chromatography (Florisil, 100–200 mesh, Aldrich) or recrystallization (**3**, **5**, **9**-endo, **10**-endo, **17**-endo). Unlike simple *N*-sulfonylimines,[3,4] **3–4** proved sensitive to hydrolysis by adventitious water and could not be purified by chromatography without extensive loss of material. Cycloadducts with endo:exo ratios of 5:1 or less were separated chromatographically and independently characterized fully. Cycloadducts with endo:exo ratios of 11:1 or greater were separated and the major diastereomer was characterized fully. Endo:exo diastereomer ratios were established spectroscopically (^1H NMR) as detailed in the supplementary material.

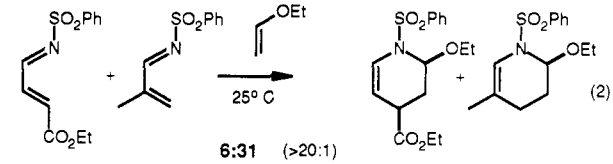
(10) (a) Characteristic coupling constants of the endo cycloadducts: C-2 OR substituent: coupling between C2-H and C3-H_{ax} J = 1.2–2.3 Hz; coupling between C2-H and C3-H_{eq} J = <1–2.7 Hz; coupling between C4-H and C3-H_{ax} J = 5.5–7.6 Hz; coupling between C4-H and C3-H_{eq} J = 1.2–2.5 Hz. C-2 aryl substituent: coupling between C2-H and C3-H_{ax} J = 3.7–5.1 Hz; coupling between C2-H and C3-H_{eq} is <1 Hz; coupling between C4-H and C3-H_{ax} J = 6.8–7 Hz; coupling between C4-H and C3-H_{eq} is <1 Hz. (b) Full details of the studies conducted are presented in supplementary material.

Table I

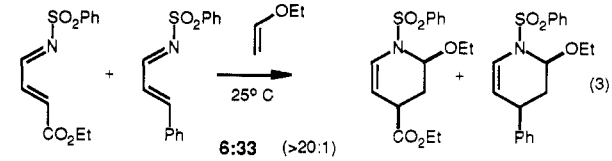
diene	dienophile (equiv)	conditions	product, endo:exo (% yield)
3	**18**, R = CH_2Ph (5)	21 °C, 46 h, CH_2Cl_2	**5**, >20:1 (88)
3	**19**, R = Et (5)	21 °C, 46 h, CH_2Cl_2	**6**, >20:1 (82)
4	**19**, R = Et (5)	21 °C, 56 h, CH_2Cl_2	**7**, >20:1 (73)
3	**20**, R = Me (3)	21 °C, 37 h, CH_2Cl_2	**8**, 2.2:1 (93)
3	**21**, R = Ph (2.5)	21 °C, 61 h, CH_2Cl_2	**9**, 5:1 (61)
3	**22** (2.5)	21 °C, 45.5 h, CH_2Cl_2, 13.3 kbar	**10**, 11:1 (48)
3	**23**, R = Me (4)	21 °C, 69 h, CH_2Cl_2	**11**, >20:1 (48)
3	**23**, R = Me (2)	21 °C, 45.5 h, CH_2Cl_2, 13.3 kbar	**11**, >20:1 (50)
3	**24**, R = Ph (2.5)	21 °C, 49.5 h, CH_2Cl_2, 13.3 kbar	**12**, 2.2:1 (42)
3	**25** (5)	0 °C, 82 h, CH_2Cl_2	**13**, >20:1 (56)
3	**26** (3)	21 °C, 49.5 h, CH_2Cl_2, 13.3 kbar	**14**, >20:1 (42)
3	**27** (3)	21 °C, 135 h, CH_2Cl_2	**15**, 2.4:1 (71)
3	**27** (2.5)	21 °C, 49.5 h, CH_2Cl_2, 13.3 kbar	**15**, 2.2:1 (74)
3	**28**, R = H (5)	21 °C, 46 h, CH_2Cl_2	**16**, >20:1 (63)
3	**29**, R = Me (2.5)	21 °C, 47.5 h, CH_2Cl_2, 13.3 kbar	**17**, 4:1 (60)

biguously established with the single-crystal X-ray structure determinations of **9**-endo, **14**-endo, and **17**-endo in conjunction with deliberate epimerization and interconversion studies (Scheme II, supplementary material).

The [4 + 2] cycloaddition reactions of **3**–**4** were found to proceed predominantly or exclusively (2.2:1 to >20.1) through an endo transition state independent of the size of the *N*-sulfonyl substituent (R^1 = Ph = CH_3). Like observations made in earlier studies,[3,4] the reactions of **3**–**4** with simple vinyl ethers (**18**, **19**, and **25**), cis-1,2-disubstituted vinyl ethers possessing a small C-2 substituent (CH_3 or OAc, **23** and **26**), and unsubstituted styrenes (**22** and **28**) proceed with high (11:1, **22**) or near exclusive (>20:1, **18**–**19**, **23**,**25**–**26**, **28**) endo diastereoselectivity. In contrast to the endo-specific cycloaddition reactions of our earlier *N*-sulfonyl aza dienes,[3,4] the reactions of **3**–**4** with trans-1,2-disubstituted dienophiles (**20**–**21**, **27**, **29**) and a cis-1,2-disubstituted vinyl ether possessing a large C-2 substituent (Ph, **24**)[11] proceed predominantly (2.2–5:1) through an endo transition state but provide significant amounts of the exo addition products. Consistent with expectations, the endo diastereoselectivity decreases with increasing reaction temperature and increases with increasing reaction pressure. As in prior studies, computational studies support the expected observation of the endo diastereoselectivity and the observed regioselectivity.[12] In addition and as a consequence of the boat transition state for the [4 + 2] cycloaddition reaction, the lone pair on nitrogen and the σ C–O bond of the dienophile lie trans periplanar to each other in the preferred endo transition state suggesting a n/σ^* stabilization of the endo transition state comparable to that responsible for the anomeric effect. A similar stabilizing n/σ^* interaction is not present in the exo [4 + 2] cycloaddition transition state, and this difference may further contribute to the unusually high endo diastereoselectivity observed in the Diels–Alder reactions of such systems.[3,4] The [4 + 2] cycloaddition reactions were found to exhibit little solvent dependency on the reaction rate [k_{rel} (**3**): CH_3CN (0.9) > CH_2Cl_2 (1) > C_6H_6 (1)][13] and were found to proceed with preservation of the dienophile stereochemistry in the stereochemistry of the reaction products. Further characteristic of a concerted Diels–Alder reaction, trans-1,2-disubstituted dienophiles were found to react more rapidly than cis-1,2-disubstituted dienophiles [for **3**, $k(E)/k(Z)$ = 13.4 (1 atm) with 1-ethoxypropene].[14] Most impressively, the *non*-complementary C-4 addition of an electron-withdrawing group to the *N*-(phenylsulfonyl)-1-aza-1,3-butadiene was found to substantially accelerate[12] the rate of [4 + 2] cycloaddition [k(**3**)/k(**30** or **32**) > 20] (eqs 2–3).[15] As such,



3 (2 equiv) **30** (2 equiv) **6** **31**



3 (2 equiv) **32** (2 equiv) **6** **33**

the aza dienes **3**–**4** were found to be sufficiently reactive to participate in intermolecular [4 + 2] cycloaddition reactions with a full range of dienophiles[16] including ketene acetals, substituted vinyl ethers, *cis*- and *trans*-2-(benzyloxy)vinyl acetate, and the relatively unreactive alkenes **22**, **28**–**29** (k(**28**)/k(**22**) > 20) (Scheme I). Notably, even the styrenes and *cis*- or *trans*-2-(benzyloxy)vinyl acetate provide a single cycloaddition regioisomer in which the inherent regioselectivity of the [4 + 2] cycloaddition reaction is unaltered by the diene C-4 ethoxycarbonyl group and the room-temperature, endo-specific reaction of **28** is consistent with the diene participation in a $LUMO_{diene}$-controlled Diels–Alder reaction. Applications of the [4 + 2] cycloaddition reactions of *N*-sulfonyl-1-aza-1,3-butadienes are in progress and the results of such studies will be reported in due course.

Acknowledgment. This work was assisted through the financial support of the National Institutes of Health (CA 42056) and a David E. Ross Fellowship (Purdue University, TC, 1990-1992). We thank Professor F. W. Fowler for stimulating discussions and for sharing the results of unpublished efforts in progress in his laboratory (ref 5). The single-crystal X-ray structure determinations of **9**-endo,

(11) The reduced endo selectivity of **24** is not surprising and may be attributed to the substantially increased destabilizing steric interactions present in the endo transition state due to the dienophile large C-2 substituent.

(12) Computational studies (AM1)[4] suggest a strong, stabilizing diene C-2/dienophile *O*R secondary orbital interaction and a predictable rate acceleration with introduction of the azadiene C-4 electron-withdrawing (CO_2Et) substituent [$\Delta\Delta E$ $HOMO_{dienophile}/LUMO_{diene}$ = –0.6 eV].[4]

(13) The solvent rate study was conducted in deuterated solvents and monitored by ^1H NMR (300 or 500 MHz) where the comparison of the amount of starting material to product could easily be determined. A solution of **3** in solvent was cooled to 0 °C and treated with ethyl vinyl ether (5 equiv).

(14) A solution of **3** (CH_2Cl_2, 0 °C) was treated with a mixture of *cis*- and *trans*-1-ethoxypropene (2.8:1, 20 equiv) and stirred while gradually warming to 21 °C. After 44 h, a 4.8:1 ratio of cycloadducts arising from *trans*- and *cis*-1-ethoxypropene, respectively, was observed by ^1H NMR (300 MHz, $CDCl_3$).

(15) A solution of **3** (0.16 mmol) and diene **30** or **32** (0.16 mmol) in CH_2Cl_2 was cooled to 0 °C and treated with ethyl vinyl ether (0.08 mmol). Inspection of the crude product by ^1H NMR (500 MHz, $CDCl_3$) showed a >20:1 (**6**:**31** or **33**) ratio of products after 52 h.

(16) Diene **3** failed to react with methyl acrylate and 1,4-benzoquinone under the conditions detailed herein.

14-endo, and 17-endo were conducted by Dr. P. Fanwick of the Purdue University X-ray crystallography facility.

Supplementary Material Available: Full experimental details for the preparation of 3-4, representative experimental procedures for the Diels-Alder reactions, full physical and spectroscopic characterization of 3-17, ^{1}H NMR spectra of 3-17, a summary of NOE difference experiments, a summary of the interconversion/epimerization studies (Scheme II), and details of the X-ray structure determinations of 9-endo, 14-endo, and 17-endo (91 pages). Ordering information is given on any current masthead page.

Tandem Free-Radical Ring Expansion and 5-*exo-dig* 5-Hexynyl Radical Cyclization: A Useful Approach to Fused Bicyclic Carbocycles

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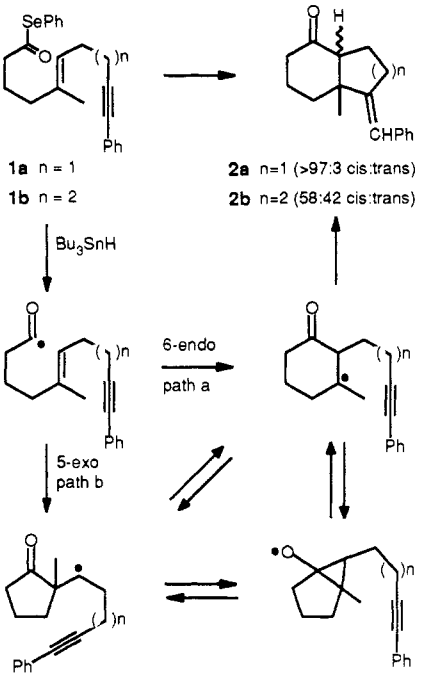
Summary: The effective preparation of hydrindan-1,4-diones, hydroazulene-1,4-diones, and hydrocyclopentacyclooctene-1,4-diones through implementation of an efficient tandem free-radical ring expansion, 5-*exo-dig* 5-hexynyl radical cyclization is detailed.

In recent studies, we have shown that acyl radicals[1] generated from phenyl selenoesters participate in effective intramolecular,[2] intermolecular,[3] macrocyclization,[4] and tandem[5] alkene addition reactions at rates greater than that of tri-*n*-butyltin hydride hydrogen abstraction (reduction)[6] and decarbonylation[7] reactions. In the course of these studies, we observed clean polycyclization of the acyl radicals generated from phenyl selenoesters **1a-b** to provide **2a-b** initiated with clean 6-*endo-trig* versus 5-*exo-trig* 5-hexenoyl radical cyclization (Scheme I). Based on past efforts, this preference for 6-*endo-trig* versus 5-*exo-trig* free-radical cyclization may be attributed to kinetic deceleration of 5-*exo-trig* cyclization due to the C-5 olefin substituent,[8] acceleration of 6-*endo-trig* cyclization[8,9]

Scheme I



(radical stability), and/or thermodynamic equilibration of initial cyclization products (5-*exo-trig* → 6-*endo-trig*).[10]

The intramolecular addition of alkyl or aryl radicals to a carbonyl group has been demonstrated to be an effective process for acyl group transfer[11] and in cases where the

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