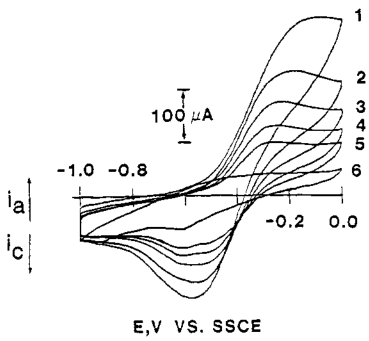

Figure 2. Cyclic voltammograms 1–5 represent five successive scans of the same electrode for which the infrared spectra are shown in Figure 2. Cyclic voltammogram 6 was recorded after the electrode had been potentiostated at 0.0 V for 7 min. The supporting electrolyte solution was 0.3 N $LiClO_4$ in methanol with a SSCE used as the reference electrode. The scan rate for each cyclic voltammogram was 50 mV/s, and the surface area was about 5 cm^2.

rivatized electrodes by transmission FT–IR spectroscopy. Doping silicon results in an increase in conductivity and a decrease in its ability to transmit infrared light. By examining silicon wafers of various doping levels, it was found that those with ~0.1-Ω cm resistivities[9] have a low enough doping level to permit useful transmission infrared studies while maintaining sufficient conductivity to permit cyclic voltammetry.[10,11] This technique allows the combination of a structurally sensitive spectroscopic probe with in situ electrochemical studies.[15]

Spectrum A of Figure 1 is of a silicon electrode modified with a polymer-bound molybdenum dinitrogen complex prepared from 2 equiv of poly-P_1, 1 equiv of poly-P_3, and 1 equiv of $Mo(N_2)_2(PPh_2Me)_4$ as described above. The infrared bands at 1943 and 1668 cm^{-1} are assigned to the ν_{N_2} and ν_{CO} stretching modes of the N_2 ligand and amide group, respectively. The absence of a strong infrared absorption at 1925 cm^{-1} indicates the polymer does not contain a significant amount of residual $Mo(N_2)_2(PPh_2Me)_4$. The remaining infrared spectra of Figure 1 and the cyclic voltammograms of Figure 2 were recorded for this same electrode immersed in 0.3 N $LiClO_4$/methanol solution. The quasi-reversible *i–E* waves of Figure 2 correspond to the oxidation of Mo(0) to Mo(I). The intensity of the amide infrared band at 1668 cm^{-1} is not decreased under these same conditions, confirming that polymer is not lost from the electrode surface. In addition by observing the N_2 infrared band of a polymer-coated electrode immersed in electrolyte solution, it can be observed that the unoxidized polymer is stable for several hours on the electrode surface. These two observations indicate that it is the oxidized form of the dinitrogen complex that is unstable. A similar oxidation of $Mo(N_2)_2(dppe)_2$ (where dppe is 1,2-bis(diphenylphosphino)ethane in a homogeneous solution results in loss of N_2 via a dissociative mechanism with a half-life of 2-3 s.[16] Presumably a similar process is taking place for the polymer-bound dinitrogen complexes. This accounts for the decrease in the peak current of oxidation and reduction waves with each successive cyclic voltammogram and the corresponding decrease in the intensity of the ν_{N_2} absorption (Figure 1). The correlation between the infrared spectra and cyclic voltammograms in Figures 1 and 2 illustrate that FT–IR spectrocopy can be a valuable probe of reactions occurring on modified silicon electrodes. In addition, a dinitrogen complex has been attached to an electrode surface whose solution analogue is capable of forming ammonia in a cyclic manner.[4,17]

Acknowledgment. This work was supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Chemical Sciences Division of the U.S. Department of Energy, under Contract EG-77-C-01-4042. We thank Dr. A. Czanderna for use of the FT–IR instrument and J. Webb for instruction in its use. Helpful discussions with J. C. Smart, C. J. Curtis, and W. L. Bell are also gratefully achnowledged.

Registry No. 1, 16605-03-1; **2**, 82638-88-8; **3**, 71734-62-8; poly-P_1, 82638-83-3; poly-P_2, 82638-85-5; poly-P_3, 82638-87-7; $Mo(N_2)_2(PPh_2Me)_4$, 66008-09-1; Si, 7440-21-3; Mo, 7439-98-7; poly(methacryloyl chloride), 26937-45-1.

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[9] We have found resistivities of commercially available silicon wafers to be only a rough guide. A suitable wafer should have a transmittance of 25–45% at 2000 cm^{-1}. A typical electrode with such a transmittance and constructed as described in footnote 10 should have a resistance of 20–80 Ω.

[10] Silicon electrodes were constructed by making an ohmic contact near the edge of a 25-mm diameter silicon wafer (111 orientation, (0.25–0.4 mm thick) with indium solder. A 25-mm glass tube was then attached with epoxy to the back of each silicon wafer. This electrode can then be mounted in an electrochemical cell by using a threaded adapter facing a KBr disk mounted in a similar manner. This configuration allows an IR light beam to pass through the center of the working electrode.

[11] Spectroelectrochemical studies in the far infrared region of the spectrum have been made previously by an internal reflection technique at Ge electrodes (ref 12), by using specular reflectance at Pt electrodes (ref 13), and by using a gold minigrid (ref 14).

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[16] Elson, C. M. *Inorg. Chim. Acta* **1976**, *18*, 209.

[17] A report of a SnO_2 electrode modified with a molybdenum dinitrogen complex containing two diphosphine ligands has appeared (ref 18), but protonation of this complex does not result in the formation of ammonia (ref 5).

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Cyclohexenones by the Barton Fragmentation of Tertiary Alcohols Derived from Furanone/Alkene Photoadducts

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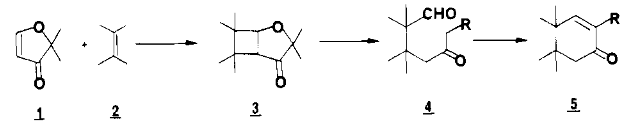
In previous publications we have described the irradiation of 2,2-dimethyl-3(2*H*)-furanone (**1**) with various alkenes **2** and the



conversion of the photoadducts **3** to a variety of synthetically useful intermediates.[2] In the particular case of forming cyclohexenones **5** from **3**, the two published methods involve rather circuitous sequences that incorporate either a Baeyer–Villiger reaction or Beckmann fragmentation in the elaboration procedure. Although the material yields are generally good, the number of discrete synthetic steps involved in each route detracts from the overall viability of the method. We now report an alternative procedure that is more efficient in terms of material yields and synthetic steps and that thus makes the overall method more attractive from a synthetic point of view.

The tertiary alcohols **6** derived from **3** are ideally disposed for an oxidative fragmentation of the C_2–C_3 bond by virtue of the

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[1] (a) Paper No. 10 in the series of photochemical annelations. For 9 see: Baldwin, S. W.; Fredericks, J. E. *Tetrahedron Lett.* **1982**, *23*, 1235. (b) Financial support from the National Institutes of Health (GM 26266) is acknowledged with appreciation.

[2] (a) Baldwin, S. W.; Wilkinson, J. M. *Tetrahedron Lett.* **1979**, 2657. (b) Baldwin, S. W.; Crimmins, M. T. *Ibid.* **1978**, 4197. (c) For a review of synthetic aspects of enone/alkene photoadditions see: Baldwin, S. W. *Org. Photochem.* **1981**, *5*, 123.

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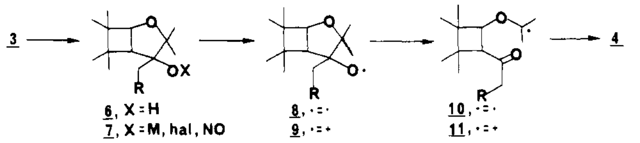
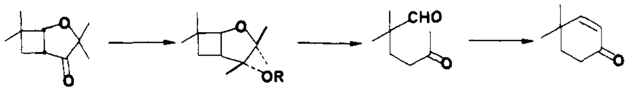


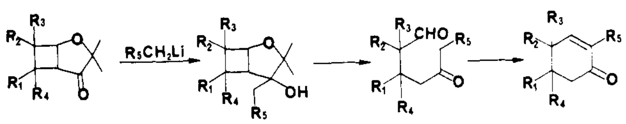
considerable stability of radical **10** (one-electron oxidation) or oxonium ion **11** (two-electron oxidation). In fact, one can imagine a variety of methods of initiate such a process, many of which have been previously employed to promote oxidative fragmentations of alcohols. Such methods include high-valent metals[3] (Ce^{4+}, Pb^{4+}, Cr^{6+} [4]), hypohalites (hypochlorites,[5] hypobromites[6]), hydroperoxides,[7] nitrite esters,[8] etc.

The requisite alcohols **6** were conveniently prepared by the addition of organolithium or organomagnesium reagents to the carbonyl group of photoadducts **3**. The products were generally homogeneous crystalline solids, presumably the result of nucleophilic attack at the less hindered exo face of the carbonyl group in yields of 71–99% (Table I). The results obtained from **12**, the photoadduct of furanone **1** and isobutylene, are illustrative. Alcohol **13** was formed in 97% yield by treatment with methyllithium.[9] Initial attempts at effecting the desired fragmentation were discouraging, the products being either recovered starting material (CrO_3, $Pb(OAc)_4$, AgTFA/Br_2, NBS/pyridine, *t*-C_4H_9OCl, H_2O_2/H_2SO_4, variously activated Me_2SO reagents) or the result of overoxidation (ceric ammonium nitrate (CAN), HOCl/HOAc). It is convenient to attribute the low level of reactivity observed for the first set of these reagents to the highly hindered nature of tertiary alcohol **13**, although several of these methods have been successfully used for the cleavage of other hindered tertiary alcohols (see, e.g., ref 3–7). The rapid overoxidation observed with CAN is not surprising in view of the documented high rates of oxidation of cyclobutanols observed with this reagent by Trahanovsky.[10] When this spectrum of reactions was applied to other tertiary alcohols from Table I, similar results were obtained, the occasional hints of success being neither uniform nor reproducible.



On the other hand, a pentane solution of nitrite ester **14** smoothly underwent the desired fragmentation reaction when irradiated with a medium-pressure lamp through Pyrex at 0 °C. The product keto aldehyde **15** (~100%), isolated after concentration of the pentane solution (*no aqueous workup*), was then smoothly cyclodehydrated (TsOH/benzene) to give 4,4-dimethylcyclohexenone (**16**) in 89% yield from **12** after distillation. In fact, similar treatment of a series of other furanone/alkene photoproducts (MeLi, NOCl, *hν*, aldol) gave related results, the overall yields for the conversion of alkenes to cyclohexenones being 62–86% (Table I).[11] These efficiencies make this annelation process attractive when compared to the more traditional methods for accomplishing similar transformations. Although it is not expected that this technique will supplant the Robinson annelation or Diels–Alder reactions, for certain substrate types it would appear to offer real advantages.

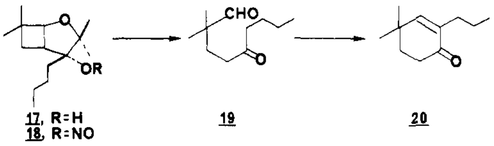
Table I. Conversion of Furanone Photoadducts 3 to Cyclohexenones

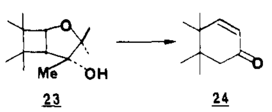


ketone[a]					alcohol, R_5 (%)	cyclohexenone[b] (%)
R_1	R_2	R_3	R_4	(%)		
H	CH_3	CH_3	H	(95)[c]	13, H (97)	16 (93)[d]
H	CH_3	CH_3	H	(85)	17, Pr (71)	20 (85)
H	CH_3	CH_2Bu	H	(95)[e]	21, H (99)	22 (93)
CH_3	CH_3	CH_3	H	(97)[c]	23, H (96)	24 (74)[f]
$(CH_2)_4$		H	H	(85)[e]	25, H (94)	26 (80)[g]
$(CH_2)_3$		H	H	(99)[c]	27, H (92)	28 (88)[h]
$(CH_2)_4$		CH_3	H	(99)[c,i]	29, H (96)	30 (92)[j]
$(CH_2)_3$		CH_3	H	(88)[k]	31, H (99)	32 (98)
H	$(CH_2)_4$		H	(81)[l]	33, H (96)	34 (78)

[a] Prepared by irradiating a hexane solution of the alkene (3–8 equiv) and 1 with a Hanovia 450-W lamp through Pyrex. [b] Obtained by exposure of crude keto aldehyde 4 to *p*-TsOH/C_6H_6/reflux. [c] Reference 17. [d] Reference 18. [e] Reference 2b. [f] Reference 19. [g] Reference 20. [h] Reference 21. [i] Photoproduct is a 4:1 mixture of HT and HH regioisomers. [j] Reference 22. [k] Photoproduct is a 9:1 mixture of HT and HH regioisomers. [l] Reference 1a.

Several points about the fragmentation process require comment. First, in many cases it is important that the irradiation be carried out in a solvent such as pentane rather than the more commonly employed benzene. In our hands, the nitrite esters derived from cycloalkene photoadducts often led to significant amounts of recovered tertiary alcohol if the fragmentation were conducted in benzene. The origin of this difficulty is obscure. The nitrite esters were formed by passing NOCl gas[12] through a pyridine solution of the tertiary alcohol at −20 °C, followed by extraction with pentane. Best results were obtained if the crude pentane extracts, after being washed with dilute HCl and dried over K_2CO_3, were directly irradiated without removal of the solvent. Products of hydrogen abstraction from the hydrocarbon solvent were not observed. It is interesting that irradiation of nitrite ester **18**, formed by the addition of *n*-butyllithium to **12** (71%) followed by nitrosation led only to keto aldehyde **19** and thus cyclohexenone **20** (85%), with no indication of products derived from λ-hydrogen abstraction from the butyl side chain. This observation attests to the great facility of the fragmentation process. Furthermore, even extremely hindered alcohols were observed to undergo the nitrosation/fragmentation process with ease. For instance, alcohols **23** and **29**[13] were smoothly converted





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(3) An excellent review of metal ion mediated oxidative fragmentations has been published. Trahanovsky, W. S. In "Methods in Free Radical Chemistry"; Huyser, E. S., Ed.; Marcel-Dekker, New York, 1973; Vol. 4, pp 133–169.

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(9) All new compounds gave satisfactory elemental analyses and spectral data consistent with the assigned structures.

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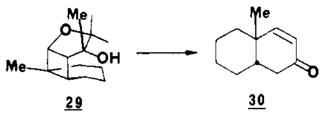
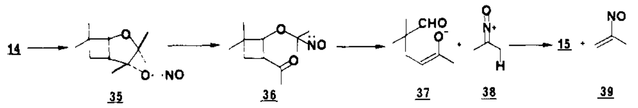
(11) The exo-alcohol isomer of **12**, formed by LiAlH reduction of the exo epoxide, also led to keto aldehyde **15** on nitrosation followed by irradiation.

(12) A slight modification of the literature procedure for generating NOCl gas was employed. Morton, J. R.; Wilcox, H. W. "Inorganic Syntheses", Bailor, J. C., Ed.; McGraw Hill: New York, 1953; Vol. IV, p 48.

to the cyclohexenones **24** and **30** in yields of 74% and 92%, respectively, on nitrosation/irradiation followed by cyclodehydration.



Factors that facilitate Barton fragmentation[8] include stabilization of the derived carbon radical, particularly by oxygen,[14] as well as strain or steric compression in the initial alkoxy radical.[15] In the context of the present work, the mechanism outlined in eq 1 (**14** → **15**) is consistent with the available information.[16]



The preceding discussion has presented an interesting and useful method for the elaboration of furanone/alkene photoadducts to cyclohexenones. The high material yields and regiochemical preferences of the photoaddition step when combined with the overall ease and efficiency of the fragmentation/cyclodehydration process recommends the reaction sequence as a general annelation technique.

Registry No. 3 ($R_1 = R_4 = H$, $R_2 = CH_3$, $R_3 = CH_2Bu$), 82555-03-1; **3** ($R_1 = R_2 = R_3 = R_4 = CH_3$), 82555-04-2; **3** ($R_1 = R_2 = (CH_2)_4$, $R_3 = R_4 = H$), 70147-91-0; **3** ($R_1 = R_2 = (CH_2)_3$, $R_3 = R_4 = H$), 70147-90-9; **3** ($R_1 = R_2 = (CH_2)_4$, $R_3 = CH_3$, $R_4 = H$), 82555-05-3; **3** ($R_1 = R_2 = (CH_2)_3$, $R_3 = CH_3$, $R_4 = H$), 82555-06-4; **3** ($R_1 = R_4 = H$, $R_2 = R_3 = (CH_2)_4$), 82526-75-8; **12**, 70147-92-1; **13**, 82555-07-5; **14**, 82555-08-6; **15**, 13544-11-1; **16**, 1073-13-8; **17**, 82555-09-7; **18**, 82555-10-0; **19**, 82555-11-1; **20**, 82555-12-2; **21**, 82555-13-3; **22**, 82555-14-4; **23**, 82555-15-5; **24**, 56140-62-6; **25**, 82555-16-6; *cis*-**36**, 55999-54-7; *trans*-**26**, 18317-63-0; **27**, 82555-17-7; **28**, 82555-18-8; **29**, 82555-19-9; **30**, 66964-45-2; **31**, 82555-20-2; **32**, 82555-21-3; **33**, 82555-22-4; **34**, 14523-53-6.

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(13) Alcohol **29** is formed by the addition of methyllithium to a minor HT photoadduct between furanone **1** and methylcyclohexene, which was separated by chromatography.

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(16) The necessary byproduct of this mechanism is 2-nitrosopropene (**39**), a previously unknown species. We have as yet been unable to detect **39** or any other C_3H_5NO species (IR, NMR, GC/MS), nor has it been possible to trap **39** by reaction with either furan or cyclopentadiene. Several nitrosoalkenes have been independently generated by base treatment of α-chlorooximes, and in the presence of reactive cyclic 1,3-dienes they often undergo a variety of interesting cycloaddition reactions. In our hands, when **39** was formed by this route, similar trapping was not observed. (a) Faragher, R.; Gilchrist, T. L. *J. Chem. Soc., Chem. Commun.* **1976**, 581. (b) Faragher, R.; Gilchrist, T. L. *J. Chem. Soc., Perkin Trans. 1* **1979**, 249. (c) Viehe, H. G.; Merenyi, R.; Francotte, E.; Van Meerssche, M.; Germain, G.; Declercq, J. P.; Bodart-Gilmont, J. *J. Am. Chem. Soc.* **1977**, *99*, 2340. (d) Hobold, V. W.; Prietz, U.; Pretzkow, W. *J. Prakt. Chem.* **1969**, *311*, 260.

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(20) Compound **26** was a varying mixture of cis and trans isomers due to isomerization at the aldehyde bearing carbon. cis Isomer: Muhle, H.; Tamm, Ch. *Helv. Chim. Acta* **1962**, *45*, 1475. trans Isomer: Mertes, M. P.; Hanna, P. E.; Ramsey, A. A. *J. Med. Chem.* **1970**, *13*, 125.

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Sonochemical and Electrochemical Synthesis of Tetramesityldisilene

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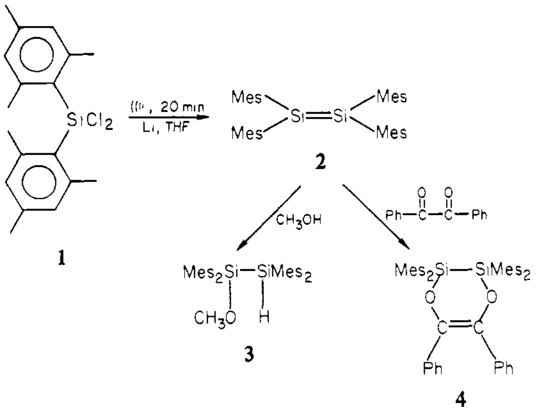
We have been investigating the effects of sonic waves on heterogeneous reactions, and we have observed some noteworthy rate enhancements.[1-4,7,10,16] For example, the Wurtz-type coupling of organic halides, RX (R = alkyl, aryl, benzyl, and benzoyl; X = Cl, Br, and I),[1] and organometallic chlorides, R_3MCl (R = alkyl, aryl; M = Si, Sn),[2] using lithium wire at room temperature proceeds at a convenient rate only in the presence of sonic waves. We have also found that sonication of a dioxane solution of α,α'-dibromo-*o*-xylene in the presence of zinc provides easy access to *o*-xylylene, a reactive intermediate that readily undergoes cycloaddition reactions to activated olefins,[3] and that ultrasound accelerates the Reformatsky reaction[4] requiring neither freshly prepared zinc powders[5] nor acid catalysts.[6] Significant rate enhancements of lithium aluminum hydride reductions of aryl halides,[7] the Barbier reaction,[8] the synthesis of thio amides,[9] and the catalytic reductions of olefins and ketones to hydrocarbons[10] point to considerable potential for sonic waves in synthesis.

In our earlier paper on the sonically accelerated couplings of silicon and tin halides[2] we reported the reaction of lithium with some simple dichlorosilanes to give high yields of cyclopolysilanes:

$$R_2SiCl_2 \xrightarrow[Li]{(((} (R_2Si)_n \qquad 70\text{–}95\%$$
$$R = Me,\ n = 6$$
$$R = Ph,\ n = 4$$

Prompted by the recent discovery by West et al.[11] that the silicon–silicon double bond can be stabilized by four mesityl groups, we extended our study to dimesityldichlorosilane (**1**). When a solution of **1** in tetrahydrofuran (THF) was irradiated with ultrasonic waves in the presence of lithium wire, a yellow color was produced immediately, and within 20 min all of **1** and most of the lithium were consumed. Tetramesityldisilene (**2**) was isolated from the product mixture in ~90% crude yield. Purification by



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(12) In our hands, decomposition of **2** was extensive when sublimation or column chromatography was attempted, frustrating our efforts to remove traces of material containing Si–H and Si–OH linkages.

0002-7863/82/1504-4992$01.25/0