(s, 3 H, CH_3), 1.28–2.80 (complex multiplet, 17 H), 3.71 (m, 4 H, NCH_2, CHOH); ^{13}C NMR CH_3 carbons at δ 32.3, 31.6, CH_2 carbons at 56.7, 43.2, 41.4, 37.3, 35.8, 33.2, 29.4, 22.7, 20.3, CH carbons at 73.0, 38.4; C carbons at 180.9, 56.5, 34.3.

Anal. Calcd for $C_{16}H_{27}NO$: C, 77.06; H, 10.91; N, 5.62. Found: C, 77.24; H, 11.04; N, 5.52.

Acetylation of 7. To a stirred solution of 78 mg (0.31 mmol) of imino alcohol **7**, 10 mL of dry tetrahydrofuran and 2 mL of pyridine under an inert atmosphere was added 1 mL of acetyl chloride in 5 mL of dry tetrahydrofuran. The resulting mixture was stirred for 5 h and quenched with 10 mL of water. The resulting solution was extracted twice with 10-mL portions of methylene chloride and the organic fractions were combined and washed twice with 10-mL portions of 10% aqueous hydrochloric acid. The organic layer was dried over anhydrous potassium carbonate, filtered, and evaporated to give a solid residue. Recrystallization from ether:ligroin (1:1) provided 76 mg (85% yield) of colorless crystals melting at 172–174 °C: IR ($CHCl_3$) C=O 5.91 (s), amide C=O 6.04 μm (s); ^1H NMR ($CDCl_3$) δ 1.00 (s, 3 H, CH_3), 1.30 (s, 3 H, CH_3), 1.35–2.65 (complex multiplet, 20 H), 1.95 (s, CH_3CO), 3.33 (m, 3 H, CH_2NCH); ^{13}C NMR CH_3 carbons at δ 34.4, 24.3 (2C), CH_2 carbons at 58.3, 45.9 (2C), 36.9, 34.9, 23.2, 21.7, 20.2, CH carbons at 62.9, 43.7, C carbons at 214.3, 169.3, 46.9, 36.3.

Anal. Calcd for $C_{18}H_{29}NO_2$: C, 74.18; H, 10.03; N, 4.81. Found: C, 74.13; H, 10.03; N, 4.81.

Lithium Aluminum Deuteride Reduction of Imino Ketone 2a and Subsequent Acetylation. A solution containing 520 mg (2.10 mmol) imino ketone **2a** in 10 mL of dry tetrahydrofuran was added to a stirred mixture of 0.2 g of lithium aluminum deuteride in 50 mL of dry tetrahydrofuran under an inert atmosphere. The reaction mixture was stirred for 2 h at room temperature and excess reducing agent was destroyed with water. The precipitate was filtered and washed well with tetrahydrofuran. The filtrate was dried over anhydrous potassium carbonate and filtered, and the solvent was removed. Recrystallization of the residue from ether provided 411 mg (79% yield) of white crystals: ^1H NMR ($CDCl_3$) δ 1.01 (s, 3 H, CH_3), 1.20 (s, 3 H, CH_3), 1.28–2.80 (complex multiplet, 17 H), 3.71 (t, 2 H, J = 8.0 Hz, CH_2N), 4.05 (bs, 1 H, OH). All ^{13}C NMR resonances of **7** are present except a methine resonance at 73.0 ppm.

Acetyl chloride (2 mL in 5 mL of dry tetrahydrofuran) was added to a stirred solution of 275 mg (1.10 mmol) of the deuterio imino alcohol above in 5 mL of pyridine and 10 mL of tetrahydrofuran. The reaction mixture was stirred for 0.5 h and water was added. The mixture was then extracted 3 times with methylene chloride and the organic extracts were combined and washed twice with 10-mL portions of 10% hydrochloric acid. The organic layer was dried over anhydrous potassium carbonate and filtered, and the solvent was removed. After chromatography on alumina, 244 mg (76% yield) of pure material was obtained: ^1H NMR ($CDCl_3$) δ 1.00 (s, 3 H, CH_3), 1.30 (s, 3 H, CH_3), 1.35–2.65 (complex multiplet, 20 H), 1.95 (s, CH_3CO), 3.35 (dd, 2 H, J = 3.0, 12.0 Hz, CH_2N). All ^{13}C NMR resonances of **8** are present except a methine resonance at 62.9 ppm.

Methylation of 7. Alcohol **7** (195 mg, 0.78 mmol) was dissolved in 25 mL of dry benzene containing 1 mL of methyl iodide, and the solution was stirred for 16 h. Precipitation of the *N*-methyl immonium salt occurred during this time. Filtration and drying in vacuo provided 297 mg (97% yield) of **9** (which melted at 282–284 °C dec) after recrystallization for ethanol:ligron (5:1).

Anal. Calcd for $C_{17}H_{30}NOI$: C, 52.13; H, 7.73; N, 3.58. Found: C, 52.30; H, 7.81; N, 3.40.

The immonium salt **9** (386 mg, 0.97 mmol) was dissolved in 25 mL of 10% potassium hydroxide solution. After being stirred for 0.5 h, the solution was extracted twice with methylene chloride and the combined organic solutions were dried over potassium carbonate. The solvent was removed to give a white solid that was recrystallized from ether:ligroin (1:1) to provide 237 mg (91% yield) of **10** as colorless crystals melting at 280–282 °C dec: IR ($CHCl_3$) C=O 5.92 μm (s); ^1H NMR ($CDCl_3$) δ 1.11 (s, 6 H, $3CH_3$), 1.14–2.40 (complex multiplet, 16 H), 2.24 (s, 3 H, NCH_3), 2.58 (d, 1 H), 2.82–3.61 (m, 3 H, $CHNCH_2$).

Acknowledgment. We thank the National Science Foundation for partial support of this work and for funds from the National Science Foundation Instrumentation Program to assist the purchase of the carbon NMR spectrometer.

Registry No. **1d**, 67505-54-8; **2a**, 91631-77-5; **2a**-d_2, 91631-78-6; **4**, 91631-79-7; **5**, 91631-80-0; **7**, 91631-81-1; **7**-d_2, 91631-82-2; **8**, 91631-83-3; **8**-*d*, 91631-84-4; **9**, 91631-85-5; **10**, 91631-86-6.

Synthesis of Enantiomerically Pure Alkyl and Aryl Methyl Sulfoxides from Cholesteryl Methanesulfinates[1a]

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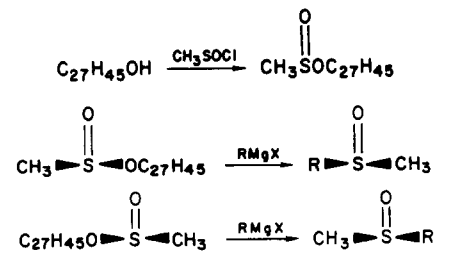
Optically active sulfoxides, chiral by virtue of asymmetry at sulfur, play a central role in organic sulfur stereochemistry especially in studies of nucleophilic substitution at sulfur.[2] Recently chiral sulfoxides have assumed importance in asymmetric synthesis.[3-5]

Simple alkyl aryl and diaryl sulfoxides are usually accessible in high enantiomeric purity, often 100%, but dialkyl sulfoxides are not. Johnson and co-workers synthesized several dialkyl sulfoxides from alkyl aryl sulfoxides by treating the latter with alkyllithiums, a reaction which interchanges the *S*-aryl and alkyllithium groups.[6] Recently, Kjaer and Malver described synthetic routes to alkyl methyl sulfoxides of high enantiomeric purity via hydrodeamination of enantiomerically homogeneous ω-aminoalkyl sulfoxides.[7] We report an alternative synthesis of methyl sulfoxides.

Alkyl aryl and diarly sulfoxides are usually prepared by treating epimerically pure crystalline menthyl arenesulfinates with the appropriate alkyl or aryl Grignard reagent.[2] For synthesis of dialkyl sulfoxides, the required alkanesulfinates have not been available epimerically pure at sulfur; e.g., the menthyl methanesulfinates are oils and attempts to separate them have not succeeded. We found, however, that substitution of cholesterol for menthol leads to crystalline cholesteryl methanesulfinates which can be separated by crystallization and which, upon treatment with alkyl or aryl Grignard reagents, yield alkyl or aryl methyl sulfoxides of high enantiomeric purity.



(1) (a) Taken in part from the Ph.D. Theses of J.D. (1974) and J.O'B. (1968). (b) University of New Hampshire. (c) Polish Academy of Sciences.

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Table I. Synthesis of Optically Active Sulfoxides, MeS(O)R, from Cholesteryl Methanesulfinates and RMgX[a]

sulfinate				sulfoxide		
no.	$[\alpha]_D$[b]	confign at S	R	yield,[c] %	$[\alpha]_D$	lit. $[\alpha]_D$
1	+77.35	*R*	*n*-Pr[d]	32	−139.0 (*c* 0.83, EtOH)	+134.4 (*c* 3, EtOH)[e]
2	−113.00	*S*	*n*-Bu[f]	52	+110.3 (*c* 1.54, Me_2CO) +109.9 (*c* 1.53, EtOH)	−116 (*c* 1.9, Me_2CO)[g]
3	−113.00	*S*	*i*-Bu[h]	50	+138.0 (*c* 0.97, EtOH)	
4	+77.35	*R*	*p*-Tol[i]	35	+148.0 (*c* 1.02, EtOH)	+156 (EtOH)[j]
5	−111.85	*S*	$PhCH_2$[k]	36	+106.0 (*c* 0.72, EtOH)	+96 (EtOH)[l]

[a] All reactions were carried out by using 0.7–1 mmol of O-cholesteryl methanesulfinate and 2.1–3 mmol of ethereal solution of Grignard reagent. [b] In benzene solution. [c] Yields after chromatography. [d] n^{20}_D 1.4662. [e] Reference 7, *S* enantiomer. [f] n^{20}_D 1.4690. [g] Reference 7, *R* enantiomer. [h] n^{20}_D 1.4632. [i] mp 71–73 °C, lit. mp 74.–74.5 °C (Cram, D. J.; Day, J.; Rayner, D. R.; von Schriltz, D. M.; Duchamp, D. J.; Garwood, D. C. *J. Am. Chem. Soc.* **1970**, *92*, 7369–7384. [j] Mislow, K.; Axelrod, M.; Rayner, D. R.; Gotthardt, H, Coyne, L. M.; Hammond, G. S. *J. Am. Chem. Soc.* **1965**, *87*, 4958–4859. [k] mp 59–61 °C. [l] Reference 8.

Addition of methanesulfinyl chloride to a solution of cholesterol and triethylamine in ether gave a quantitative yield of the epimeric cholesteryl methanesulfinates. Fractional crystallization yielded samples of the two epimers in 3.5% and 0.7% yield. Presumably these yields could be improved by more effective recrystallization techniques or by the use of chromatography. Treatment of each epimer with various Grignard reagents gave the sulfoxides listed in Table I.

By comparison with literature rotations (see Table I), it can be seen that our methyl sulfoxides prepared from our methanesulfinates are of high enantiomeric purity. (*S*)-Methyl *n*-propyl sulfoxide prepared by Kjaer and Malver was believed by them to be enantiomerically pure,[7] but when our rotation of 139° is used, their sample is calculated to be 97% pure (entry no. 1). On the other hand our (*R*)-methyl *p*-tolyl sulfoxide, prepared from the same (*R*)-methanesulfinate as our methyl *n*-propyl sulfoxide, is calculated to be 95% enantiomerically pure (entry no. 4). Our (*S*)-methyl *n*-butyl sulfoxide is calculated to be 95% pure based on 116° as its maximum rotation[7] (entry no. 2). (*S*)-Benzyl methyl sulfoxide was prepared by Mislow from menthyl phenylmethanesulfinate.[8] Benzyl *p*-tolyl sulfoxide produced by him from his ester was calculated to be 91% enantiomerically pure based on the sulfoxide's highest reported rotation. Assuming that Mislow's reported 96° rotation refers to a 91% enantiomerically pure methyl benzyl sulfoxide, the calculated value for the pure sulfoxide is 105°. This leads in turn to the conclusion that our (*S*)-methanesulfinate is enantiomerically pure rather than 95% pure (entry no. 5).

We were not able to determine the enantiomeric purity of *n*-butyl methyl sulfoxide or isobutyl methyl sulfoxide by means of ^1H NMR spectroscopy using a chiral shift reagent[9] or by the Pirkle method[10] since the methyl singlets overlapped with multiplets from the hydrogens α to the sulfinyl groups of the butyl radicals. A mixture of (+)-(*R*)-methyl *p*-tolyl sulfoxide, $[\alpha]$ +145.5° (*c* 0.4, acetone) and tris[3-(*tert*-butylhydroxymethylene)-*d*-camphorato]-europium exhibited an ^1H NMR spectrum with only one S-methyl singlet although the enantiomeric purity of the sulfoxide with such an optical rotation is equal to 86.8%.[4,11] Fraser used tris[3-(heptafluoropropylhydroxymethylene)-*d*-camphorato]europium for the quantitative determination of the enantiomeric purity of a sample of benzyl methyl sulfoxide, $[\alpha]$ +86° (*c* 0.8, EtOH).[12] Integration of the two methyl peaks, which were separated by 5.2 Hz, showed that the major enantiomer comprised 94.5% of the mixture. Thus, the enantiomeric purity of this sulfoxide was 89% and the calculated rotation of pure sulfoxide 96.6°. This purity is about 7% higher than the purity calculated by using the value of 105.5° based on Mislow's data.[8] According to this value, the purity of the sulfoxide with $[\alpha]$ +86° is 82%.

Thus, from these conflicting results it is not possible to determine exactly the enantiomeric purities of the (*R*)- and (*S*)-methanesulfinates, but these esters doubtless lead to sulfoxides of 95% or greater enantiomeric purity.

Experimental Section

General Methods. Infrared spectra were taken with a Perkin-Elmer 137 infrared spectrophotometer in CCl_4 solution. ^1H NMR spectra were recorded with a JEOL MH-60 spectrometer in benzene solution with Me_4Si as the internal standard. Chemical shifts are given in δ (ppm). Melting points, determined with a capillary melting point apparatus, and boiling points are uncorrected. Diethyl ether was distilled from lithium aluminum hydride immediately before use. Petroleum ether (50–60 °C), pentane, and ligroin were distilled from sodium. Methylene chloride was distilled from phosphorus pentoxide and acetone from potassium carbonate. Methanesulfinyl chloride was prepared by the method of Douglass[13] in 86% yield: bp 56 °C (28 mmHg) (lit.[13] bp 48 °C (22 mmHg)); n^{20}_D 1.5004 (lit.[13] n^{25}_D 1.5038). Cholesterol was a commercial product (Aldrich): mp 147–149 °C; $[\alpha]_D$ −40.05° (*c* 2, chloroform).

Synthesis of (−)-Cholesteryl (−)-(*S*)-Methanesulfinate and (−)-Cholesteryl (+)-(*R*)-Methanesulfinate. (−)-Cholesterol (38.6 g, 0.100 mol) and triethylamine (10.1 g, 0.100 mol) were dissolved in ether (380 mL) in a 1-L three-necked round-bottomed flask equipped with an efficient mechanical stirrer, addition funnel, and calcium chloride drying tube. A solution of methanesulfinyl chloride (9.8 g, 0.10 mol) in ether (50 mL) was added dropwise with vigorous stirring at 0 to 5 °C. An immediate white precipitate was formed. After completion of the addition (1.5 h), the mixture was stirred at 0–5 °C for an addition 2 h. Then, more ether and water (100 mL) were added. The ethereal layer was removed and washed successively with 5% cold HCl solution, 2% sodium bicarbonate solution, and water and then dried over magnesium sulfate. After evaporation of the ether, *O*-cholesteryl methanesulfinate was obtained as a white solid (43 g, 99%): mp 97–112 °C; $[\alpha]_D$ −30.82 (benzene). After seven recrystallizations 1.5 g of pure (−)-cholesteryl (−)-(*S*)-methanesulfinate was isolated: $[\alpha]_D$ −113.95° (benzene); mp 123–124 °C; IR 1057 cm^{-1}; ^1H NMR (benzene) δ 2.25 (s, 3 H, CH_3S), 0.7–2.8 (m, 45 H, protons of the cholesteryl skeleton). Anal. Calcd for $C_{28}H_{48}SO_2$: C, 74.94; H, 10.78; S, 7.14. Found: C, 74.52; H, 10.76; S, 7.03.

The recrystallizations proceeded as follows: no., solvent (by volume), $[\alpha]_D$ (benzene), mp, yield (g). 1st, A:E:PE (8:1:1), −55.56°, 110–117 °C; 16 g. 2nd, A:E:P (3:8:1), −69.50°, 113–119 °C 12 g. 3rd, A:E:P (1:6:10), −83.10°, 115–118 °C, 9 g. 4th, A:P (1:8), −87.15°, 115–118 °C, 7 g. 5th, A:P (1:8), −98.70°, 123–124 °C, 5.2 g. 6th, P:L (5:1), −110.20°, 123–124 °C, 3.5 g. 7th, P:L (5:2),

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-113.95°, 123-124 °C, 2.5 g. 8th P:L (5:2), -113.95°, 123-124 °C, 1.5 g. A = acetone, E = ethyl ether, PE = petroleum ether (50-60 °C), P = pentane, L = ligroin.

The mother liquor from the first recrystallization yielded 27 g of ester, $[\alpha]_D$ -11.32°, which was recrystallized from 25 mL of ether to yield 8 g of ester, $[\alpha]_D$ -29.85°. The mother liquor yielded 19 g of ester, $[\alpha]_D$ -2.52°, mp 80-82 °C. This was recrystallized 5 times to yield 0.3 g of *R* ester: $[\alpha]_D$ +77.35°; mp 115.5-116.5 °C. The IR and NMR spectra were identical with those of the (-)-(*S*) diastereomer. Anal. Calcd for $C_{28}H_{48}O_2$: C, 74.94; H, 10.78; S, 7.14. Found: C, 74.65; H, 10.58; S, 7.09. These five recrystallizations proceeded as follows. 1st, A, -0.70°, 81-91 °C, 15.2 g, 2nd, A:E (6:5), +10.23°, 85-95 °C, 8 g. 3rd, A:E (1:1), +38.50°, 98-105 °C, 2 g. 4th, A:E (2:1), +70.45°, 112.5-113.5 °C, 1.2 g, 5th, A:E (1:1), +77.35°, 115.5-116.5 °C, 0.3 g. 6th, A:E (1:1), +76.85°, 116.0-116.5 °C, 0.2 g.

Synthesis of Optically Active Sulfoxides: General Procedure. A solution of *O*-cholesteryl methanesulfinate (1 equiv) in ether (25.0 mL/mmol of sulfinate) was added dropwise to an ethereal solution of the appropriate Grignard reagent (3 equiv) at room temperature. After completion of the addition, the reaction mixture was stirred for 2 h at room temperature. Then, the reaction mixture was quenched with 50 mL of a saturated aqueous solution of ammonium chloride. After being stirred the layers were separated and the ether phase was extracted twice with water (20 mL). The combined water solution after saturation with sodium chloride was extracted with chloroform (5 × 20 mL). The chloroform solution was dried over magnesium sulfate. Rotoevaporation of the solvent left the crude sulfoxide which was purified by column chromatography on aluminum oxide using methylene chloride as eluent. IR spectra were the same as those of authentic samples of the racemic sulfoxides. The yield and optical rotation of sulfoxides obtained in this way are collected in Table I.

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Registry No. MeS(O)Cl, 676-85-7; (*R*)-MeS(O)Pr, 37177-70-1; (*S*)-MeS(O)Bu, 763-95-1; (*S*)-MeS(O)-*i*-Bu, 26451-17-2; (*R*)-MeS(O)-*p*-C_6H_4Me, 1519-39-7; (*S*)-MeS(O)CH_2Ph, 14090-81-4; (-)-cholesterol, 57-88-5; (-)-cholesteryl (-)-(*S*)-methanesulfinate, 63520-69-4; (-)-cholesteryl (+)-(*R*)-methanesulfinate, 63520-66-1.

A Novel Reaction of Cyanamide with 1,3-Diketones

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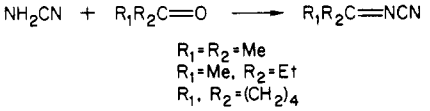
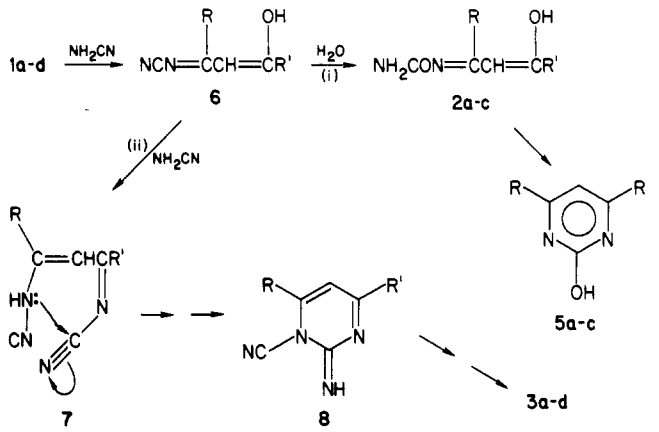
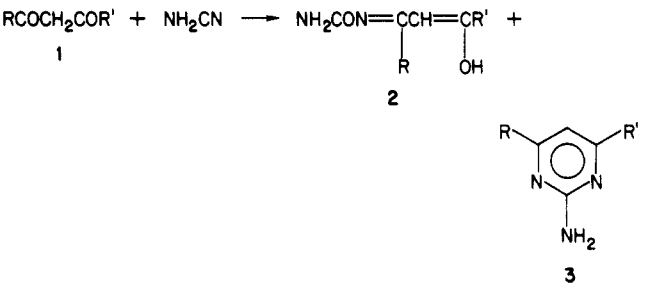
Only a few reactions of cyanamide with ketones have been reported in the literature. Typical examples are the reactions of simple aliphatic ketones with cyanamide at 60 °C for 6 h to give the corresponding cyanoimino compound in moderate yield[1] and the reaction of sodium cyanamide with ethyl acetoacetate to give the same derivative of the keto group.[2]



Scheme I



We have found that cyanamide and 2,4-pentanedione (**1a**) react in water without added acid or base to give after 8 h, a 23% yield of 4-[(aminocarbonyl)imino]-2-penten-2-ol (**2a**) and a 38% yield[3] of 2-amino-4,6-dimethylpyrimidine (**3a**). The same reaction in methanol gave much lower



a, R = R' = Me
b, R = CF_3; R' = Me
c, R = CF_3; R' = *i*-Pr
d, R = Me; R' = Ph

yields of **2a** and **3a**. The reaction in aqueous carbonate gave a 43% yield[3] of **3a** and 3% of 4-amino-3-penten-2-one (**4**).

The reaction of cyanamide with four other 1,3-diketones has also been explored. Most of these reactions were run for several days at room temperature and only the major products were isolated. (1) When a 1.25 M aqueous solution of 1,1,1-trifluoropentane-2,4-dione (**1b**) was treated with a 1 molar excess of cyanamide, a 68% yield[3] of 2-amino-4-(trifluoromethyl)-6-methylpyrimidine (**3b**) was obtained. Under similar conditions a 1:1 molar ratio of **1b** and cyanamide gave a good yield of a mixture of **3b** and 4-(trifluoromethyl)-2-hydroxy-6-methylpyrimidine (**5b**). No intermediates for this reaction could be detected by TLC. (2) With water, in which it was not very soluble, 1,1,1-trifluoro-5-methylhexane-2,4-dione (**1c**) gave only a very low yield of 4-(trifluoromethyl)-2-hydroxy-6-isopropylpyrimidine (**5c**) with cyanamide. In homogeneous media, methanol or 50/50 methanol/water, a 25% yield of **5c** was isolated. (3) From benzoylacetone and 3 equiv of cyanamide, a 13% yield[3] of 2-amino-4-methyl-6-phenylpyrimidine (**3d**) was isolated. (4) With 1,3-cyclohexanedione there was little or no reaction.

Information about the reaction mechanism was obtained from control experiments. Thus, urea, a possible hydrolysis product of cyanamide,[4] did not react with **1a** under the reaction conditions. Furthermore, cyanoguanidine, the dimerization product of cyanamide,[4] did not react with **1a**

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(3) All yields reported for **3** are based upon a stoichiometry of 2 mol of cyanamide to 1 mol of ketone 1.
(4) American Cyanamide Company, "Cyanamide", New York, 1958.

0022-3263/84/1949-4072$01.50/0