Cleavage of Isoxazolines with Tricarbonyltris(acetonitrile)molybdenum and Silica Gel. Synthesis of 1-(2-Oxoalkyl)cyclopropanols from Isoxazoline-5-spirocyclopropanes

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An efficient synthesis of 1-(2-oxoalkyl)cyclopropanols [or (1-hydroxycyclopropyl)methyl ketones] from isoxazoline-5-spirocyclopropanes by selective N–O cleavage effected by $Mo(CO)_3(MeCN)_3$ and silica gel is described.

Recently we described the synthesis and rearrangement of isoxazoline- and isoxazolidine-5-spirocycloalkanes.[1] The easy thermal cleavage of the N–O bond combined with the strain relief due to the cyclopropane or cyclobutane ring opening rendered possible some useful transformations which were employed for the synthesis of a number of heterocyclic structures present in many alkaloids.[2]

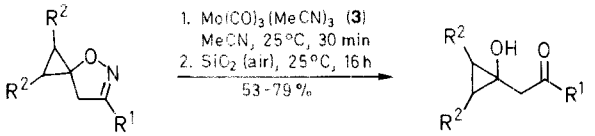
With the aim of expanding the synthetic use of isoxazoline-5-spirocyclopropanes **1**, we investigated the feasibility of new routes, other than thermal and photolytic ones, for the N–O bond cleavage and the obtainment of different reaction pathways. We now report a new synthesis of 1-(2-oxoalkyl)cyclopropanols [or (1-hydroxycyclopropyl)methyl ketones)] **2a–e** via reaction of compounds **1a–e** with molybdenum carbonyl complexes.

The reductive N–O cleavage of isoxazolines in the presence of hexacarbonyl molybdenum has been investigated under thermal conditions.[3–5] This process is reported to occur via an intermediate $Mo(CO)_5$-isoxazoline complex in which the possible delocalization of a π-d electron from the metal to the π^* (LUMO) orbital of the C=N–O moiety is responsible for the weakening and the facile cleavage of the N–O bond.[4] When 3,5-diarylisoxazolines were heated in wet acetonitrile with 1 molecular equivalent of $Mo(CO)_6$, several products were formed which were derived from N–O and (C-4)–(C-5) bond cleavages.[5] On the other hand, high yields of β-hydroxyketones were obtained from 3,5-disubstituted isoxazolines by a similar procedure, using a modified work-up of the reaction mixture.[3]

On the basis of these findings we expected that, in principle, several reactions are possible when the above mentioned procedures are applied to isoxazoline-5-spirocyclopropanes. One possibility is that in the molybdenum carbonyl-complexed isoxazoline only the N–O bond is cleaved to give, after hydrolysis, cyclopropanols. Other possibilities are the cleavages of both the N–O and the (C-1)–(C-2) cyclopropane bond with formation of reduced open-chain compounds.

The clean and selective transformation of isoxazoline-5-spirocyclopropanes **1a–e** into 1-(2-oxoalkyl)cyclopropanols **2a–e** (Scheme A) can be achieved by adding compounds **1** to a solution of $Mo(CO)_3(MeCN)_3$ **(3)** prepared by heating hexacarbonylmolybdenum **(4)** in boiling anhydrous acetonitrile for 4 h.[6,7] After 30 min at room temperature, the reaction mixture is placed on a silica gel PTLC plate which is then allowed to stand for 16 h. Elution of the TLC plate with ethyl acetate affords compounds **2a–e** in > 95% purity. Under these conditions, the conversion of compounds **1** is complete and selective and products **2** are obtained in 53–79% yields (Table 1). For preparations on a larger scale, the procedure was modified by using powdered silica gel instead of the TLC plate. The use of silica gel is essential for the conversion **1** → **2**. Indeed, if the reaction mixture of **1a** and **3** is quenched with water after 24 h at room temperature, only the starting material **1a** is recovered by PTLC. Exposure of the reaction mixture to air also seems to play an important role. Thus, treatment of the above reaction mixture with silica gel and water for several hours (without exposure to air), followed by elution, gave cyclopropanol **2a** in only 30% yield, contaminated with unidentified molybdenum complexes. Conversion of **1a** is still incomplete and the yield of **2a** is poor when the reaction conditions are modified either by reducing the air exposure time to 1.5 h, or by using a 1:1 molecular ratio of **1a:4**.

By using $Mo(CO)_3(MeCN)_3$, a more reactive complexing agent than $Mo(CO)_6$,[7,9] the complexation of isoxazolines **1** can be achieved under very mild conditions, and mild conditions are also employed in the reduction, decomplexation, and hydrolysis steps which proceed on silica gel and lead to selective N–O bond cleavage. Under these conditions, the sensitive 1-hydroxycyclopropyl compounds **2a–e** can be isolated. Earlier related attempts[10] to cleave the heterocyclic ring of isoxazolino[4,5]-fused cyclopropanes without affecting the cyclopropane ring were unsuccessful.



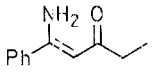
1a-e **2a-e**

1, 2	R^1	R^2	R^2
a	Ph	H	H
b	Ph	$(CH_2)_4$	
c	$PhCH_2$	H	H
d	n-C_6H_{13}	H	H
e	$(MeO_2C)_2CH(CH_2)_2$	H	H

Scheme A

Application of the reported method using $Mo(CO)_6$[3,5] (Method E) to the conversion described here gave much poorer results. Thus, isoxazoline **1a** was converted (on a 1 mmol scale) to a mixture of 1-hydroxy-1-phenacylcyclopropane (**2a**; 18%), 1-amino-1-phenyl-1-penten-3-one (**5**; 18%), and unreacted starting material (**1a**; 25%).

Compound **5** is also isolated in 13% yield when the method of Ref. 5 is applied to isoxazoline **1a**; its formation clearly results from N–O bond and cyclopropane (C-1)–(C-2) bond cleavages.



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The hydroxycyclopropyl moiety is an interesting target in view of its further transformation into cyclobutane derivatives.[11] The present procedure for the synthesis of 1-(2-oxoalkyl)cyclopropanols **2** is general; the substituents at C-3 of the isoxazolines **1** can be various alkyl or aryl groups. Compounds **1** are obtained by cycloaddition of nitrile oxides with commercial or easily available methylenecyclopropanes.[12] The present method compares favorably with other synthetic approaches, as evidenced by comparison of our synthesis of **2a** with a reported[8] synthesis which requires at least six steps. With the aim of applying our method to other isoxazolines, we studied the reactions of 3,5-diphenyl-4,5-dihydroisoxazole **(6)** and of *cis*-3a,5,6,6a-tetrahydro-3-phenyl-4*H*-cyclopent[*d*]isoxazole **(7)** with the carbonylmolybdenum complexes **3** and **4** (Scheme **B**).

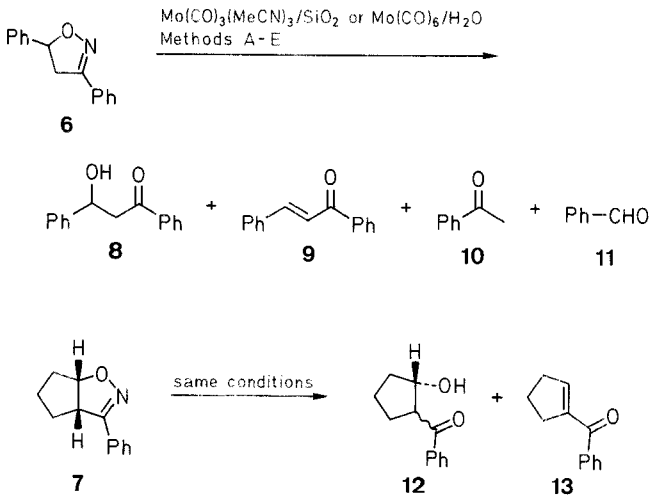
Treatment of isoxazoline **6** with $Mo(CO)_3(MeCN)_3$ **(3)** at room temperature afforded 3-hydroxy-1,3-diphenyl-1-propanone **(8)** and benzylidenacetophenone **(9)**, in addition to unreacted starting material **(6)** in varying amounts depending on the work-up (Table 2, entries 1–3). The best result (39% of **8**; entry 2, Method B on a 3 mmol scale) is achieved by pouring the reaction mixture of **6** and **3** on a glass disk containing 20 g of silica gel and exposing it to the air for 16 h. Retroaldolization occurs to a limited extent, only 4% of acetophenone being observed in the crude mixture. With Method E (the method of Ref. 3; Table 2,

Table 1. 1-(2-Oxoalkyl)cyclopropanols **2** Prepared

Product	Yield[a] (%)	mp (°C)	Molecular Formula[b]	MS (70 eV)[c] *m/z* (%)	IR ($CDCl_3$)[d] ν_{OH}; $\nu_{C=O}$ (cm^{-1})	^{1}H-NMR ($CDCl_3$)[e] δ, J(Hz)	^{13}C-NMR ($CDCl_3$)[f] δ ($\underline{C}HR^2$–$\underline{C}HR^2$, $\underline{C}H_2$–C=O, C–OH, C=O)
2a[g]	72	55–56	$C_{11}H_{12}O_2$ (176.2)	176 (M^+, 3), 147 (10), 120 (10), 105 (100)	3550, 1680	0.47–0.55 (m, 2H); 0.85–1.0 (m, 2H); 3.33 (s, 2H); 3.9 (br, 1H); 7.4–7.6 (m, 3H); 7.9–8.0 (m, 2H)	12.75, 45.90, 52.02, 200.66
2b	73	64–65	$C_{15}H_{18}O_2$ (230.3)	230 (M^+, 2), 147 (16), 120 (65), 105 (100)	3545, 1685	1.0–1.35 (m, 8H); 1.8–2 (m, 2H); 3.35 (s, 2H); 3.8 (br, 1H); 7.45–7.65 (m, 3H); 7.95–8.0 (m, 2H)	19.74, 38.70, 58.01, 200.52
2c	59	oil	$C_{12}H_{14}O_2$ (190.2)	190 (M^+, 0.2), 134 (1.5), 118 (23), 91 (100)	3550, 1710	0.65–1.05 (m, 4H); 2.70 (s, 2H); 3.45 (br, 1H); 3.70 (s, 2H); 7.1–7.5 (m, 5H)	12.63, 49.11, 51.93, 209.2
2d	79	oil	$C_{11}H_{20}O_2$ (184.3)	155 (3), 113 (22), 85 (19), 71 (10), 57 (26), 43 (100)	3450, 1720[h]	0.34–0.45 (m, 2H); 0.72–0.94 (m, 5H); 1.18–1.38 (m, 6H); 1.48–1.66 (m, 2H); 2.39 (t, 2H, J = 7.5); 2.65 (s, 2H); 3.6 (br, 1H)	12.49, 49.79, 51.87, 212.08
2e	53	oil	$C_{12}H_{18}O_6$ (258.3)	258 (M^+, 3), 229 (5), 187 (22), 171 (20), 155 (100), 113 (67)	3550, 1735, 1720	0.35–0.45 (m, 2H); 0.75–0.85 (m, 2H); 2.16 (q, 2H, J = 7.2); 2.55 (t, 2H, J = 7.2); 2.64 (s, 2H); 3.43 (t, 1H, J = 7.2); 3.71 (s, 7H)	12.73, 50.17, 50.06, 209.93

[a] Yield of isolated product obtained according to the General Procedure on a 1 mmol scale.
[b] Satisfactory microanalyses were obtained with a Perkin Elmer 240 C element analyzer: C ±0.4, H ±0.1.
[c] Obtained by GC inlet on a 5790A-5970A Hewlett-Packard instrument.
[d] Recorded on a Perkin-Elmer 283 spectrophotometer.
[e] Recorded on a Varian XR 300 (300 MHz) or on a Perkin Elmer R 32 (90 MHz) (for **2e**).
[f] Recorded on a Varian FT-80 A (20 MHz).
[g] The spectral data are in accordance with Lit.;[8] the mp was not reported.
[h] Neat.

entry 4), the yield (as estimated by ^{1}H-NMR analysis of the crude mixture) of hydroxyketone **8** is lower (16%), although conversion is complete, because of the extensive formation of benzylidenacetophenone (**9**; 20%), acetophenone (**10**; 25%), and benzaldehyde (**11**; 13%). Our results can be regarded to be satisfactory when compared with those reported in Ref. 5 (Table 2, entry 5).



Scheme **B**

Table 2. Cleavage of Isoxazolines **6** and **7** with $Mo(CO)_3(MeCN)_3$ or $Me(CO)_6$

Isoxazoline[a]	Entry	Method or Ref.	Yield (%)[b]				
			Unreacted Starting Material	Products[c]			
6				**8**	**9**	**10**	**11**
	1	A	30	37	18	2	2
	2[d]	B	27	39	27	4	–
	3	C	28	21	16	–	–
	4	E	–	16	20	25	13
	5	[5]	–	–	4	1	–
7				**12**	(*cis* : *trans*)		**13**
	6	A	48	38	(0.9)		10
	7[e]	B	52	27	(1.5)		17
	8	B[f]	41	34	(1.8)		14
	9	C	23	6	(*cis* only)		3
	10	D	45	21	(1.4)		7
	11	E	32	24	(5)		10

[a] For Methods A–E, see experimental section. In entry 5 are reported the results obtained by Kobayashi et al.[5] by reaction of the isoxazoline **6** (1 equiv) with $Mo(CO)_6$ (1 equiv) and H_2O (1 equiv) in boiling MeCN for 17 h.

[b] The yields were evaluated by ^{1}H-NMR analysis (300 MHz) of the crude mixture. All experiments were carried out with 1 mmol of isoxazoline except for entries 2, 7, 8 (3 mmol), and 10 (2 mmol).

[c] All isolated products were characterized by their ^{1}H-NMR data which were in accord with the literature data (**12** *cis* and *trans*,[13] **13**[14]).

[d] Yields of products isolated by flash chromatography (silica gel 230–400 mesh; eluent $CHCl_3$/hexane, 2 : 1): **6**, 31%; **8**, 43%; **9**, 11%.

[e] Yields of the products isolated by flash chromatography (silica gel 230–400 mesh; eluent light petroleum/EtOAc, 4 : 1): **7**, 41%; *cis*-**12**, 15%; *trans*-**12**, 15%; **13**, 14%.

[f] The work-up was carried out with 16.6 g of silica gel (70–230 mesh, for column chromatography) per mmol of isoxazoline.

The stereochemical implications of this reductive process were studied for the reaction of the isoxazoline **7** with the complex **3**. In all experiments (Table 2, entries 6, 7, 8, 10) carried out with silica gel, the conversion is 50–60%, yielding the hydroxyketone[13] **12** as a *cis*/*trans* mixture and some chalcone[14] **13**. Method E[3] (Table 2, entry 11) produces the hydroxyketone **12** in lower yield (24%), but with a higher *cis*/*trans* ratio (5 : 1). When silica gel is not used (Table 2, entry 9), only a small amount of *cis*-**12** is obtained (6%) besides ketone **13** (3%) and starting material **7** (23%). These observations could indicate an effect of silica gel on the epimerization processes which possibly occur during hydrolysis of the β-hydroxyimine intermediate, via the tautomeric enamine.[15]

In conclusion, the use of $Mo(CO)_3(MeCN)_3$ combined with the treatment with silica gel, is a useful technique to convert isoxazoline-5-spirocyclopropanes **1** into 1-(2-oxoalkyl)cyclopropanols **2** and might be useful in cases in which the reported methods of reductive isoxazoline ring cleavage (hydrogenation on Raney nickel in the presence of strong acids[15,16] or Ti^{3+} reductions[17]) cannot be used due to the presence of additional reactive functional groups. Some epimerization and α,β-unsaturated ketone formation, as observed with compounds **6** and **7**, indicate a limitation of the general applicability of this method for the cleavage of isoxazolines.

Preparative TLC plates and silica gel were purchased from Merck and were used without further treatment.

The starting isoxazoline-5-spirocyclopropanes **1a–c** and **1e** were prepared as previously described[1,2] by cycloaddition of methylenecyclopropane or methylenenorcarane (for **1b**) with nitrile oxides generated from hydroximic chlorides. The isoxazolines **6** and **7** were synthesized by reported methods.[18,19]

6-Hexyl-4-oxa-5-azaspiro[2.4]hept-5-ene (1d):

A solution of heptanal oxime[20] (4.288 g, 32 mmol), NCS (4.128 g, 32 mmol) and Et_3N (0.1 mL) in $CHCl_3$ (70 mL) is stirred for 1 h at room temperature. Then, basic alumina (pH 9.5; 41 g) is added,[21] the mixture is cooled to −40 °C and methylenecyclopropane (3.5 g, 65 mmol) is added via cannula. The vessel is then tightly stoppered and allowed to stand for 6 days at room temperature. The mixture is filtered, the solvent is evaporated, and CCl_4 (15 mL) is added at room temperature to the residual oil. The precipitated succinimide is filtered off, the filtrate is evaporated, and the residual crude oil is flash-chromatographed on silica gel (230–400 mesh; eluent cyclohexane/EtOAc, 12 : 1) to give the product **1d** as a light yellow oil; yield: 1.316 g (23%).

$C_{11}H_{19}NO$ (181.2) calc. C 72.88 H 10.56 N 7.73
found 72.61 10.48 7.71

MS (70 eV): *m/z* (%) = 181 (M^+, 1); 83 (18); 82 (30); 69 (12); 57 (10); 55 (22); 43 (100); 42 (98).

IR ($CDCl_3$): ν = 3090, 3010, 1620 cm^{-1}

^{1}H-NMR ($CDCl_3$/TMS): δ = 0.6–0.7 (m, 2H); 0.85 (t, 3H, *J* = 6.7 Hz); 1.05–1.15 (m, 2H); 1.2–1.4 (m, 6H); 1.48–1.59 (m, 2H); 2.34 (t, 2H, *J* = 6.9 Hz); 2.95 (s, 2H).

^{13}C-NMR ($CDCl_3$/TMS): δ = 11.41 t (2C); 13.81 q; 22.29 t; 26.07 t; 28.10 t; 28.71 t; 31.27 t; 41.94 t; 64.84 s; 159.71 s.

1-(2-Oxoalkyl)cyclopropanols [or (1-Hydroxycyclopropyl)methyl Ketones, 2a–e]; General Procedure:

A solution of $Mo(CO)_3(MeCN)_3$ (**3**; 2 mmol) is prepared[6,7] by heating for 4 h under N_2 a 0.33 M solution (6 mL) of $Mo(CO)_6$ (**4**) in anhydrous MeCN (distilled over CaH_2). The isoxazoline (**1a–e**; 1 mmol, neat or dissolved in the minimum amount of MeCN) is added to the solution of **3** at room temperature. The solution is stirred for 30 min, then worked up by the following procedure:

The mixture is cannulated onto a 4 cm broad strip of the PTLC plate (silica gel F254, 2 mm thickness), the plate is exposed to air for 16 h under a hood, then eluted with EtOAc. The silica gel is removed and extracted with EtOAc (100 mL). Concentration of the extract under vacuum affords the product **2a–e** in > 95% purity. Analytical samples are obtained by flash chromatography on silica gel (230–400 mesh)

using the following eluents: **2a**, hexane/EtOAc (5:2); **2b**, $CHCl_3$; **2c**, light petroleum/EtOAc (3:1); **2d**, EtOAc; **2e**, cyclohexane/EtOAc (1:2).

1-Amino-1-phenyl-1-penten-3-one (5):
Application of the method of Lit.[5] to isoxazoline **1a** leads to the formation of aminoketone **5** as an oil which is purified by flash chromatography on silica gel (230–400 mesh; light petroleum/EtOAc, 3:1 as eluent); yield: 13%.

$C_{11}H_{13}NO$ (175.2)

MS (70 eV): *m/z* (%) = 175 (M^+, 15); 146 (100).

Exact Mass (VG 70-70 EQ Instrument): calc. 175.0996, found 175.0996.

IR ($CDCl_3$): ν = 3500 (m), 3250 (w), 1635 (s), 1600 (s), 1575 (s), 1530 (s), 1490 (s) cm^{-1}.

^{1}H-NMR ($CDCl_3$/TMS, 300 MHz): δ = 1.13 (t, 3H, J = 7.5 Hz); 2.41 (q, 2H, J = 7.5 Hz); 5.2 (br, 1H); 5.43 (s, 1H); 7.35–7.75 (m, 5H); 9.9 (br, 1H).

1-Phenacylcyclopropanol (2a); 10 mmol-Scale Procedure:
The solution (70 mL of MeCN) containing complex **3** and isoxazoline **1a** is treated with silica gel (60 H for TLC; 25 g). The resultant slurry is poured on two large glass disks (18 cm diameter) which are then exposed at the air for 48 h under a hood. Then, the silica gel is poured on a Büchner funnel (5 cm diameter) containing a 2 cm layer of silica gel (70–230 for column chromatography). Elution with EtOAc (300 mL) and evaporation of the eluate under reduced pressure affords product **2a** in > 95% purity; yield: 1.06 g (60%).

Ring Cleavage of Isoxazolines 6 and 7 Using $Mo(CO)_3(MeCN)_3$ (3):
The reactions are performed as above, but different work-up procedures are used.

Method A: Same work- up as in the General Procedure.

Method B: The mixture is poured onto a large glass disk containing silica gel (6.67 g per mmol of isoxazoline, Merck 70–230 mesh for column chromatography). After 16 h of air exposure under a hood, the silica gel is poured into a Büchner funnel and eluted with EtOAc (100 mL per mmol of isoxazoline). Evaporation of the eluate under reduced pressure affords the crude mixture of products.

Method C: The mixture is treated with H_2O (1 mL per mmol of isoxazoline), stirred at room temperature for 24 h, and filtered twice through celite. Evaporation of the solvent affords the crude mixture of products.

Method D: To the mixture is added silica gel (70–230 mesh, 2 g per mmol of isoxazoline). The solvent is removed under reduced pressure (20 mbar), and the residue is poured onto a large glass disk. After 16 h of air exposure under a hood, the silica gel is poured into a Büchner funnel and eluted with EtOAc (50 mL per mmol of isoxazoline). The solvent is evaporated under reduced pressure to give the crude mixture of products.

Ring Cleavage of Isoxazolines Using $Mo(CO)_6$[3] (4):

Method E: To isoxazoline **6** or **7** (1 mmol) in MeCN (5 mL) containing H_2O (5 drops), $Mo(CO)_6$ (130 mg, 0.5 mmol) is added and the mixture is heated to reflux under N_2 for 1.5 h. Then, silica gel (2 g) is added to the cooled mixture, the solvent is evaporated under reduced pressure (20 mbar), and the residue is chromatographed on silica gel using EtOAc as eluent.

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