Ozonolyses of Cycloalkenes in the Presence of Carbonyl Compounds

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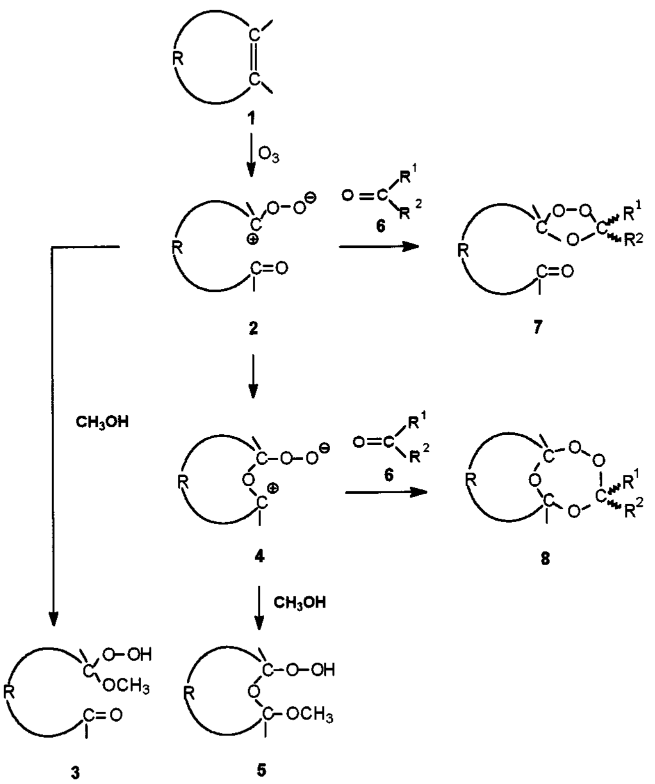
Ozonolysis reactions of a series of cyclic olefins **1** in the presence of carbonyl compounds **6** provided the corresponding cross-ozonides **42**. Further reactions of ozonides **42** with the independently prepared carbonyl oxide $^+CH_2-OO^-$ gave diozonides of structure **43**. All of the new ozonides have been isolated as pure substances and characterized by their ^{1}H- and ^{13}C-NMR spectra.

Introduction

Ozonolysis reactions of monocyclic, bicyclic, and polycyclic olefins have been studied under a variety of aspects and conditions.[1] From the results of early studies, it appeared that ozonide formation is restricted to a small number of cyclic olefins, viz. those having 4−6-membered rings.[2] By contrast, attempts to prepare ozonides by starting from larger ring systems were futile and gave peroxidic products, which, although occasionally called "in-situ ozonides", have not been proven to have a 1,2,4-trioxolane structure. More recently, however, the scope of existence of monomeric ozonides has been extended up to those derived from a 16-membered ring.[3] Ozonolysis reactions of certain cycloolefins **1** in methanol, however, revealed a partially anomalous behavior as compared to acyclic olefins.[4][5] A priori, one would have expected that the primary fragments **2** are trapped by methanol to give compounds of type **3**. However, in addition to **3**, variable amounts of the isomeric products **5** were obtained. This was explained in terms of an intramolecular reaction between the carbonyl oxide moiety and the aldehyde group of **2** to give intermediate **4**, which is subsequently trapped by methanol to give **5**.[4][5]

In the work reported here we were interested in finding out whether the postulated intermediates of types **2** and **4** can be trapped by added carbonyl compounds **6** to give ozonides of type **7** and cyclic peroxides of type **8**, respectively. In the pursuit of this goal, we ozonized unsubstituted monocyclic olefins of type **9**, methyl-substituted monocyclic olefins of type **10**, the bicyclic olefins **11** and **12**, the tricyclic unsaturates **13** and **14** and the tetracyclic unsaturates **15** and **16** in the presence of formaldehyde (**6a**), acetyl cyanide (**6b**), and benzoyl cyanide (**6c**). The ozonolyses of **9a**−**9e**, **11**, and **12** were additionally carried out in the presence of acetaldehyde (**6d**).



Results and Discussion

All reactions were performed in dichloromethane at temperatures between 0 °C and −78 °C depending on the solubility of the substrates. The molar ratio of the olefinic substrate and of the carbonyl compound **6** was in each case 1:2. The crude reaction products were analyzed by ^{1}H-NMR spectroscopy and subsequently separated by flash chromatography to isolate the peroxidic products. All yields reported are those of isolated, pure compounds.

Coozonolyses of the unsubstituted monocyclic olefins **9a**−**9e** and the carbonyl compounds **6a** and **6b**, and coozonolyses of **9a**−**9c** with **6c** and **6d** afforded, in each case, the corresponding ozonides **17** in yields between 11% and 74%. By contrast, cyclic peroxides of type **18** could only be obtained in coozonolysis reactions of **9b**−**9e** with formaldehyde and of **9b** and **9d** with acetaldehyde.

The successful coozonolyses of the cycloolefins **9** with carbonyl compounds opened a convenient short-path syn-

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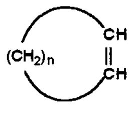

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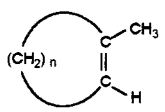

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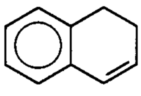

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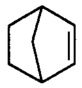

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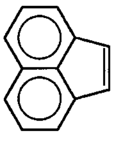
	a	b	c	d	e
n	3	4	5	6	10

	a	b
n	3	4



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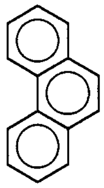

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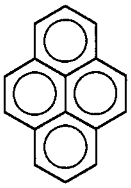

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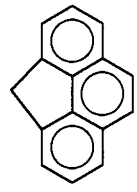

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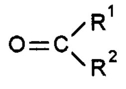
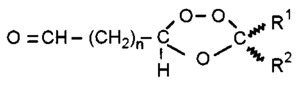


6

	a	b	c	d
R^1	H	CH_3	C_6H_5	H
R^2	H	CN	CN	CH_3



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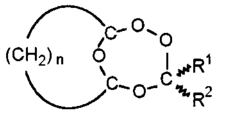

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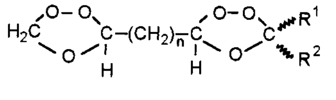
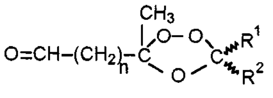


19

	structural units			yields (%) of		
	n	R^1	R^2	17	18	19
a	3	H	H	46	–	29
b	4	H	H	68	36	10
c	5	H	H	74	19	33
d	6	H	H	36	16	57
e	10	H	H	17	10	32
f	3	H	CH_3	37	–	21
g	4	H	CH_3	27	10	35
h	5	H	CH_3	17	8	10
i	6	H	CH_3	19	8	9
j	10	H	CH_3	17	–	10
K	3	CH_3	CN	47	–	19
l	4	CH_3	CN	70	–	32
m	5	CH_3	CN	61	–	53
n	3	C_6H_5	CN	42	–	25
o	4	C_6H_5	CN	62	–	26
p	5	C_6H_5	CN	33	–	34



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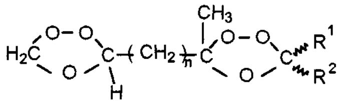

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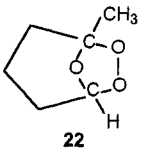


22

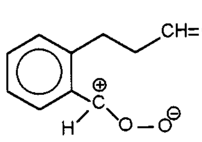
	structural units			yields(%) of		
	n	R^1	R^2	20	21	22
a	3	H	H	–	–	74
b	3	CH_3	CN	–	–	63
c	3	C_6H_5	CN	–	–	61
d	4	H	H	50	38	–
e	4	CH_3	CN	42	32	–

thesis for the hitherto unknown types of ozonides **17**, which bear aldehyde groups at the side chains. This offered the possibility to generate diozonides by cycloaddition reactions of carbonyl oxides and these aldehyde groups. To test this possibility, we have ozonized vinyl acetate – which is known to provide formaldehyde *O*-oxide – in the presence of one of the ozonides **17a**–**17p**. All of these reactions afforded the corresponding diozonides **19** and thus opened a very convenient entry to this class of compounds.

Ozonolysis of 1-methylcyclopentene (**10a**) in the presence of formaldehyde (**6a**), acetyl cyanide (**6b**), or benzoyl cyanide (**6c**) did not provide the corresponding ozonides **20**. Instead, ozonide **22** was obtained in yields of 62–74%. By contrast, ozonolysis of 1-methylcyclohexene (**10b**) in the presence of **6a** and **6b** did provide the corresponding ozonides **20**. Reactions of these ozonides with CH_2COO afforded the corresponding diozonides **21d** and **22e** in yields of 38% and 32%, respectively. The differences in the behavior of **10a** and **10b** are, given the results of previous studies, that intramolecular ozonide formation is more favored in the ozonolysis of five- rather than six-membered rings.[1a]

Ozonolysis of **11** could, a priori, generate two intermediates, viz. **23** and **24**. Co-ozonolyses of **11** and the carbonyl compounds **6a**–**6d** gave, however, only ozonides of type **25**. Since they were obtained in rather high yields, it can be concluded that ozone cleavage at **11** occurred with high preference in one direction to give intermediate **24** rather than **23**. This is surprising, since one would have expected the zwitterion in **23** to be resonance stabilized as opposed to that in **24**. Ozonolysis of vinyl acetate in the presence of one of the ozonides **25a**–**25d** provided the corresponding diozonides **26**. The fact that these diozonides were obtained in considerably higher yields than most of the diozonides of structure **19** can be ascribed to the higher reactivity of the aromatic aldehyde groups in compounds **25** as opposed to the aliphatic aldehyde groups in compounds **17**.

Ozonolyses of norbornene (**12**) in the presence of one of the carbonyl compounds **6b**–**6d** gave the corresponding



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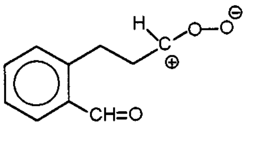

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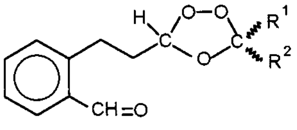

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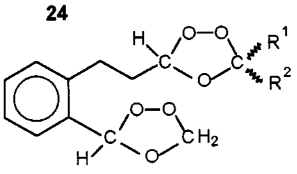
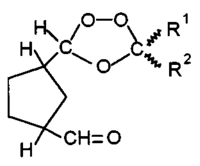


26

	structural units		yields (%) of	
	R^1	R^2	25	26
a	H	H	63	58
b	CH_3	CN	80	77
c	C_6H_5	CN	78	55
d	H	CH_3	74	69

ozonides **27**, which could be converted into the corresponding diozonides **28** by reaction with formaldehyde *O*-oxide.



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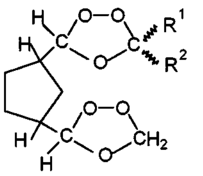
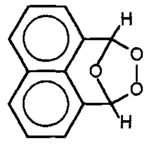


28

	structural units		yields (%) of	
	R^1	R^2	27	28
b	CH_3	CN	18	25
c	C_6H_5	CN	28	24
d	H	CH_3	48	37

Ozonolysis of the tricyclic substrate **13** in the presence of **6a**−**6c** did not provide the corresponding cross-ozonides. Instead, 16−26% of ozonide **29** and 42−60% of **30** were obtained. These observations, together with the results obtained in the attempted coozonolyses of **10a**, are in line with the experience that intramolecular ozonide formation is very favored in the ozonolysis of substrates in which the double bond is incorporated in a five-membered ring.

Ozonolysis of the tricyclic substrate **14** in the presence of **6a**−**6c** provided the corresponding cross-ozonides **31** in fair to good yields to the exclusion of the ozonide of **14**. This is again in line with the experience that intramolecular ozonide formation is not favored in the ozonolysis of substrates in which the double bond is incorporated in a six-membered ring.[1a] Reactions with formaldehyde *O*-oxide converted the monoozonides **31a**−**31c** to the corresponding diozonides **32** in yields of 63−66%.



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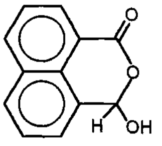

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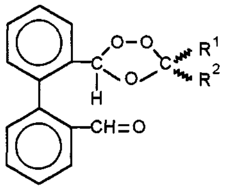

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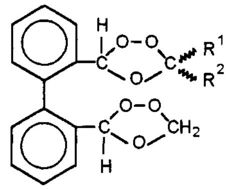
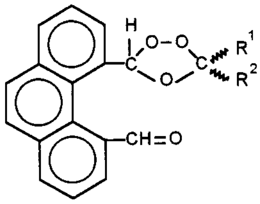


32

	structural units		yields (%) of	
	R^1	R^2	31	32
a	H	H	49	63
b	CH_3	CN	74	63
c	C_6H_5	CN	72	66

Ozonolysis of the tetracyclic substrate **15** in the presence of **6a** and of **6b** gave the corresponding cross-ozonides **33**, whereas no cross-ozonide was obtained in the ozonolysis of **15** in the presence of benzoyl cyanide (**6c**). In each case, the ozonide of **15**, viz. **35**, was a coproduct. Reactions of ozonides **33a** and **33b** with formaldehyde *O*-oxide afforded the diozonides **34a** and **34b**, respectively.



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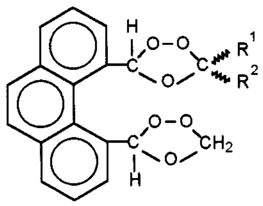

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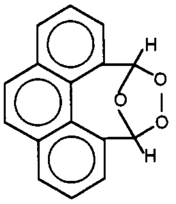


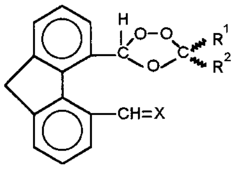
35

	structural units		yields (%) of		
	R^1	R^2	33	34	35
a	H	H	52	41	17
b	CH_3	CN	62	46	22
c	C_6H_5	CN	–	–	24

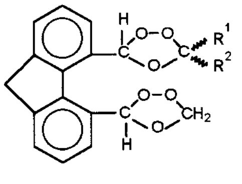
Ozonolysis of the tetracyclic substrate **16** in the presence of formaldehyde (**6a**) gave the cross-ozonide **36a**, which could be converted into diozonide **38a** by reaction with formaldehyde *O*-oxide. A by-product of the ozonolysis of **16** was the acid aldehyde **40**, which was partly converted into **41** by treatment with *O*-methylhydroxylamine. Ozonolysis of **16** in the presence of **6b** provided cross-ozonide **36b**, which was converted into ozonide **37b** by treatment of the crude reaction mixture with *O*-methylhydroxylamine and isolated as such. Since ozonide **36b** was not isolated, it could not be treated with formaldehyde *O*-oxide and, hence, diozonide **38b** was not accessible. Additional products from the ozonolysis of **16** were ozonide **39** and, once again, acid aldehyde **40**. Acid aldehydes are common by-products in the ozonolysis of fused polyaromatic hydrocarbons.[1b] Ozonolysis of **16** in the presence of **6c** did not provide cross-ozonide **36c**, but only ozonide **39**. The lack of formation of cross-ozonide **36c** in the ozonolysis of **16** and – as shown above – the lack of formation of cross-ozonide **33c** in the ozonolysis of **15** in the presence of benzoyl cyanide (**6c**) may be due to steric restraints.

All of the aforementioned cross-ozonides, diozonides, and other cyclic peroxides were isolated as pure compounds. The unsymmetrically substituted cross-ozonides and diozonides in which $R^1 \neq R^2$ were mixtures of the possible stereoisomers.

In most cases, the major isomer could be individually isolated, whereas the minor isomer could only be enriched in admixture with the major one.



36 (X = O)
37 (X = $NOCH_3$)



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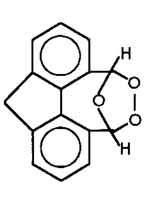

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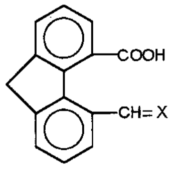
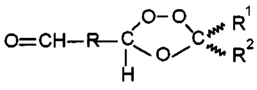


40 (X = O)
41 (X = $NOCH_3$)

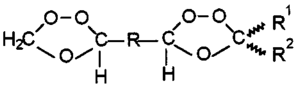
	structural units		yields (%) of				
	R^1	R^2	36[a]	37	38	39	40[b]
a	H	H	82	–	26	–	46
b	CH_3	CN	–	12	–	13	45
c	C_6H_5	CN	–	–	–	64	–

[a] Yield of recrystallized from cold dichloromethane.
[b] Yield of isolated products at room temperature.

The structures of the hitherto unknown cross-ozonides of type **42** and diozonides of structure **43** were established by ^1H- and ^{13}C-NMR spectroscopy and by reductions with triphenylphosphane. Reductions of ozonides **42** gave the fragments **44** and **45** in a ratio of ca. 1:1, while reductions of diozonides **43** gave fragments **44**, **45**, and **46** in a ratio of ca. 1:1:1.



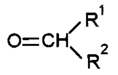
42



43

O=CH–R–CH=O

44



$O=CH(R^1)(R^2)$

45

$O=CH_2$

46

In the ^1H-NMR spectra, the ozonide moieties of the cross-ozonides **42** could be recognized by signals of the CH groups in the ozonide rings. They appeared in the range of δ = 5.0–6.0, depending on the nature of R^1 and R^2. In addition, the aldehyde groups showed their CH signals in the range of δ = 9.6–9.8. In the ^1H-NMR spectra of the diozonides **43**, the CH and the CH_2 groups of the ozonide rings showed characteristic signals in the range of δ = 5.0–6.0. In the ^{13}C-NMR spectra of the cross-ozonides **42**, the two chemically nonequivalent carbon atoms in the ozonide rings exhibited signals in the range of δ = 90 to 120, and the carbon atoms of the aldehyde groups showed signals in the range of δ = 200–205. In the ^{13}C-NMR spectra of the diozonides **43**, the ozonide rings could also be clearly recognized by signals of the two carbon atoms of the ozonide rings in the range of ca. δ = 90–120.

The *cis* and *trans* isomers have been tentatively assigned based on the assumption that, as in other reported cases,[6][7] the ^1H-NMR signal of the hydrogen atom attached to the ozonide ring appears in a higher field position for the *trans* isomer than for the *cis* isomer.

The structural assignments of the tetroxepanes **18b–18e**, **18g**, and **18i** are based on characteristic ^1H- and ^{13}C-NMR signals of the CH_2 groups and CH groups in the tetroxepane ring systems. In the ^1H-NMR spectrum the CH_2 group showed singlet signals at δ = 5.02 and at δ = 5.17, and the CH groups showed triplets at δ = 4.84 and δ = 5.12. In the ^{13}C-NMR spectrum, the CH_2 group exhibited a signal at δ = 93.71 and the CH groups gave signals at δ = 100.86 and δ = 103.15.

Conclusions

The results obtained in this study provide ample evidence that carbonyl oxides that are formed in the ozonolysis of cyclic olefins can be readily trapped by "foreign" carbonyl compounds like aldehydes or acyl cyanides to give cross-ozonides. This opens a convenient short-path synthesis for ozonides that bear aldehyde groups and are thus capable of further reactions. As one of several conceiveable aldehyde reactions, the cycloaddition with formaldehyde *O*-oxide was realized to give a variety of diozonides. This represents another new short-path synthesis for ozonides that were not known previously.

The overall result of this study is thus two-fold: (a) the demonstration that carbonyl oxides derived from the ozonolysis of cycloolefins can be trapped by added "foreign" carbonyl compounds and (b) the preparation of new types of ozonides without the necessity to prepare the parent olefins.

Experimental Section

NMR spectra: Bruker AC-300. ^{1}H-NMR and ^{13}C-NMR spectra were recorded in $CDCl_3$ (unless stated otherwise) with TMS as the internal reference. − Chromatographic separations: Flash chromatography on silica gel.

Ozonolyses and Reductions of Ozonides: Unless stated otherwise, the following procedure was used: The ozonolysis reaction was carried out in dichloromethane at −78 °C until the solution turned blue. Residual ozone was flushed out with nitrogen, the solvent was distilled off at room temperature under reduced pressure, and the residue was separated by flash chromatography. Reductions of isolated ozonides were carried out on samples of ca. 20−40 mg in ca. 0.6 mL of $CDCl_3$ with an excess of triphenylphosphane, and the products were analyzed by ^{1}H-NMR spectroscopy.

Caution: All ozonolysis reactions, chromatographic separations, and reductions of ozonides were carried out behind protective safety glass shields in a fumehood. Ozonides were invariably transported, e.g. to the analytical laboratory, in thick-walled steel containers. Safety glasses and gloves must be worn.

Ozonolysis of 9a in the Presence of 6a: Ozonolysis of 0.50 g (7.3 mmol) of **9a** and 1 mL of **6a** (freshly prepared by pyrolysis of paraformaldehyde) in 50 mL of dichloromethane at −78 °C, followed by distillation of the solvent under reduced pressure, gave a liquid residue. From this liquid, 0.49 g (3.4 mmol, 46.1%) of **17a** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

(1,2,4-Trioxolan-3-yl)butanal (17a): Colorless liquid. − ^{1}H NMR: δ = 1.66−1.77 (m, 4 H), 2.48 (t, J = 8.23 Hz, 2 H), 5.06 (t, J = 5.12 Hz, 1 H), 5.09 (t, J = 5.12 Hz, 1 H), 5.10 (s, 1 H), 9.70 (s, 1 H). − ^{13}C NMR: δ = 16.58, 30.77, 43.60, 94.41, 103.61, 202.10. − $C_6H_{10}O_4$ (146.1): calcd. C 49.31, H 6.90; found C 49.42, H 6.94. − Reduction of **17a** with TPP gave 1,5-pentanedial [δ = 1.90 (quint), 2.47 (t), 9.72 (s)].

Ozonolysis of Isopropenyl Acetate in the Presence of 17a: Ozonolysis of 0.40 g (2.8 mmol) of **17a** and 0.55 g (5.5 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.16 g (0.81 mmol, 28.9%) of **19a** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

3-[3-(1,2,4-Trioxolan-3-yl)propyl]-1,2,4-trioxolane (19a): Colorless liquid. − ^{1}H NMR: δ = 1.57−1.64 (m, 2 H), 1.76−1.82 (m, 4 H), 5.04 (s, 2 H), 5.13 (t, J = 5.12 Hz, 2 H), 5.18 (s, 2 H). − ^{13}C NMR: δ = 18.43. 30.69, 94.43, 103.64. − $C_7H_{12}O_6$ (192.2): calcd. C 43.73, H 6.29; found C 43.64, H 6.42. − Reduction of **19a** with TPP gave 1,5-pentanedial.

Ozonolysis of 9b in the Presence of 6a: Ozonolysis of 0.98 g (12.0 mmol) of **9b** and 1 mL of **6a** (freshly prepared by pyrolysis of paraformaldehyde) in 50 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.70 g (4.4 mmol, 36.4%) of **18b** and 1.31 g (8.2 mmol, 68.2%) of **17b** were isolated [solvent: dichloromethane/diethyl ether, 15:1].

7,8,10,11-Tetraoxabicyclo[4.4.1]undecane (18b): Colorless liquid. − ^{1}H NMR: δ = 1.46−1.50 (m, 4 H), 1.67−1.75 (m, 4 H), 4.84 (t, J = 5.12 Hz, 1 H), 5.03 (s, 1 H), 5.12 (t, J = 5.12 Hz, 1 H), 5.17 (s, 1 H). − ^{13}C NMR (BB): δ = 23.30, 23.33, 30.59, 33.04, 93.71, 100.86, 103.15. − ^{13}C NMR (CB): δ = 23.30 (t, J = 480 Hz), 23.33 (t, J = 510 Hz), 30.59 (t, J = 515 Hz), 33.04 (t, J = 530 Hz), 93.87 (t, J = 630 Hz), 100.95 (d, J = 640 Hz), 103.15 (d, J = 685 Hz). − $C_7H_{12}O_4$ (160.2): calcd. C 52.48, H 7.55; found C 52.37, H 7.46. − Reduction of **18b** with TPP gave 1,6-hexanedial.

(1,2,4-Trioxolan-3-yl)pentanal (17b): Colorless liquid. − ^{1}H NMR: δ = 1.48 (m, 2 H), 1.67 (m, 4 H), 2.47 (t, J = 6.14 Hz, 2 H), 5.02 (s, 1 H), 5.13 (t, J = 5.12 Hz, 1 H), 5.17 (s, 1 H), 9.75 (s, 1 H). − ^{13}C NMR: δ = 21.66, 23.19, 30.86, 43.47, 93.92, 103.29, 201.94. − $C_7H_{12}O_4$ (160.2): calcd. C 52.48, H 7.55; found C 52.37, H 7.64. − Reduction of **17b** with TPP gave 1,6-hexanedial.

Ozonolysis of Isopropenyl Acetate in the Presence of 17b: Ozonolysis of 0.48 g (3.0 mmol) of **17b** and 0.60 g (6.0 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.06 g (0.29 mmol, 9.7%) of **19b** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

3-[4-(1,2,4-Trioxolan-3-yl)butyl]-1,2,4-trioxolane (19b): Colorless liquid. − ^{1}H NMR: δ = 1.49−1.60 (m, 4 H), 1.73−1.80 (m, 4 H), 5.03 (s, 2 H), 5.13 (t, J = 5.12 Hz, 2 H), 5.18 (s, 2 H). − ^{13}C NMR: δ = 23.95, 31.34, 94.41, 103.86. − $C_8H_{14}O_6$ (206.2): calcd. C 46.60, H 6.84; found C 46.56, H 6.77. − Reduction of **19b** with TPP gave 1,6-hexanedial.

Ozonolysis of 9c in the Presence of 6a: Ozonolysis of 0.20 g (2.1 mmol) of **9c** and 1 mL of **6a** (freshly prepared by pyrolysis of paraformaldehyde) in 50 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.07 g (0.4 mmol, 19.1%) of **18c** and 0.27 g (1.6 mmol, 74.2%) of **17c** were isolated [solvent: dichloromethane/diethyl ether, 15:1].

8,9,11,12-Tetraoxabicyclo[5.4.1]dodecane (18c): Colorless liquid. − ^{1}H NMR: δ = 1.40−1.55 (m, 6 H), 1.60−1.77 (m, 4 H), 4.86 (t, J = 5.14 Hz, 1 H), 5.03 (s, 1 H), 5.13 (t, J = 5.14 Hz, 1 H), 5.20 (s, 1 H). − ^{13}C NMR: δ = 23.69, 24.07, 29.48, 31.38, 34.57, 94.41, 101.83, 104.13. −$C_8H_{14}O_4$ (174.2): calcd. C 55.16, H 8.10; found C 54.56, H 7.98. − Reduction of **18c** with TPP gave 1,7-heptanedial [δ = 1.36 (quint), 1.63 (quin.), 2.45 (t), 9.71 (s)].

(1,2,4-Trioxolan-3-yl)hexanal (17c): Colorless liquid. − ^{1}H NMR: δ = 1.38−1.47 (m, 4 H), 1.62−1.75 (m, 4 H), 2.45 (t, J = 8.24 Hz, 2 H), 5.04 (s, 1 H), 5.14 (t, J = 5.12 Hz, 1 H), 5.19 (s, 1 H), 9.77 (s, 1 H). − ^{13}C NMR: δ = 22.18, 23.93, 29.22, 31.28, 44.04, 94.40, 103.96, 202.91. − $C_8H_{14}O_4$ (174.2): calcd. C 55.16, H 8.10; found C 55.07, H 8.23. − Reduction of **17c** with TPP gave 1,7-heptanedial.

Ozonolysis of Isopropenyl Acetate in the Presence of 17c: Ozonolysis of 0.54 g (3.1 mmol) of **17c** and 0.62 g (6.2 mmol) of isopropenyl acetate in 50 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.23 g (1.03 mmol, 33.1%) of **19c** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

3-[5-(1,2,4-Trioxolan-3-yl)pentyl]-1,2,4-trioxolane (19c): Colorless liquid. − ^{1}H NMR: δ = 1.43−1.49 (m, 6 H), 1.70−1.75 (m, 4 H), 5.04 (s, 2 H), 5.14 (t, J = 5.12 Hz, 2 H), 5.20 (s, 2 H). − ^{13}C NMR: δ = 24.00, 29.50, 31.34, 94.43, 104.06. − $C_9H_{16}O_4$ (220.2): calcd. C 49.09, H 7.32; found C 48.71, H 7.28. − Reduction of **19c** with TPP gave 1,7-heptanedial.

Ozonolysis of 9d in the Presence of 6a: Ozonolysis of 0.49 g (4.5 mmol) of **9d** and 1 mL of **6a** (freshly prepared by pyrolysis of paraformaldehyde) in 50 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.13 g (0.74 mmol, 16.2%) of **18d** and 0.30 g (1.60 mmol, 35.6%) of **17d** were isolated [solvent: dichloromethane/diethyl ether, 15:1].

9,10,12,13-Tetraoxabicyclo[6.4.1]tridecane (18d): Colorless liquid. − ^{1}H NMR: δ = 1.35−1.42 (m, 8 H), 1.52−1.75 (m, 4 H), 4.84 (t, J = 5.12 Hz, 1 H), 5.05 (s, 1 H), 5.14 (t, J = 5.12 Hz, 1 H), 5.21 (s, 1 H). − ^{13}C NMR: δ = 23.76, 24.14, 29.53, 29.60, 31.45, 34.71, 94.41, 101.96, 104.19. − $C_9H_{16}O_4$ (188.2): calcd. C 57.44, H 8.57; found C 58.78, H 8.43. − Reduction of **18d** with TPP gave 1,8-octanedial [δ = 1.36 (quin.), 1.66 (t), 2.45 (t), 9.72 (s)].

(1,2,4-Trioxolan-3-yl)heptanal (17d): Colorless liquid. − ^{1}H NMR: δ = 1.32−1.40 (m, 6 H), 1.69−1.74 (m, 4 H), 2.42 (t, J = 8.24 Hz, 2 H), 5.04 (s, 1 H), 5.14 (t, J = 5.12 Hz, 1 H), 5.19 (s, 1 H), 9.77 (s, 1 H). − ^{13}C NMR: δ = 22.18, 23.93, 29.16, 29.27, 31.37, 44.15, 94.37, 104.06, 203.19. − $C_9H_{16}O_4$ (188.2): calcd. C 57.44, H 8.57; found C 57.12, H 8.69. − Reduction of **17d** with TPP gave 1,8-octanedial.

Ozonolysis of Isopropenyl Acetate in the Presence of 17d: Ozonolysis of 0.24 g (1.3 mmol) of **17d** and 0.26 g (2.6 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.17 g (0.74 mmol, 56.7%) of **19d** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

3-[6-(1,2,4-Trioxolan-3-yl)hexyl]-1,2,4-trioxolane (19d): Colorless liquid. − ^{1}H NMR: δ = 1.36−1.45 (m, 8 H), 1.70−1.74 (m, 4 H), 5.04 (s, 2 H), 5.13 (t, J = 5.12 Hz, 2 H), 5.20 (s, 2 H). −^{13}C NMR: δ = 24.04, 29.53, 31.41, 94.40, 104.11. − $C_{10}H_{18}O_6$ (234.3): calcd. C 51.26, H 7.74; found C 51.57, H 7.61. − Reduction of **19d** with TPP gave 1,8-octanedial.

Ozonolysis of 9e in the Presence of 6a: Ozonolysis of 1.0 g (6.0 mmol) of **9e** and 1 mL of **6a** (freshly prepared by pyrolysis of paraformaldehyde) in 50 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.15 g (0.62 mmol, 10.3%) of **18e** and 0.25 g (1.02 mmol, 17.1%) of **17e** were isolated [solvent: dichloromethane/diethyl ether, 15:1].

13,14,16,17-Tetraoxabicyclo[10.4.1]heptadecane (18e): Colorless liquid. − ^{1}H NMR: δ = 1.17−1.47 (m, 16 H), 1.62−1.75 (m, 4 H), 4.84 (t, J = 5.12 Hz, 1 H), 5.04 (s, 1 H), 5.12 (t, J = 5.12 Hz, 1 H), 5.19 (s, 1 H). − ^{13}C NMR: δ = 23.76, 24.07, 24.22, 29.75, 29.81, 31.48, 94.37, 102.07, 104.24. − $C_{13}H_{24}O_4$ (244.1): calcd. C 52.48, H 7.55; found C 52.37, H 7.46. − Reduction of **18e** with TPP gave 1,12-dodecanedial [δ = 1.26−1.37 (m), 1.63 (quint), 2.45 (t), 9.71 (s)].

(1,2,4-Trioxolan-3-yl)undecanal (17e): Colorless liquid. − ^{1}H NMR: δ = 1.29−1.43 (m, 14 H), 1.60−1.72 (m, 4 H), 2.41 (t, J = 8.24 Hz, 2 H), 5.01 (s, 1 H), 5.14 (t, J = 5.12 Hz, 1 H), 5.17 (s, 1 H), 9.75 (s, 1 H). − ^{13}C NMR: δ = 22.32, 23.92, 24.09, 29.41, 29.66, 31.38, 44.10, 94.22, 104.07, 202.98. − $C_{13}H_{24}O_4$ (244.1): calcd. C 52.48, H 7.55; found C 52.33, H 7.64. − Reduction of **17e** with TPP gave 1,12-dodecanedial.

Ozonolysis of Isopropenyl Acetate in the Presence of 17e: Ozonolysis of 1.44 g (5.9 mmol) of **17e** and 1.18 g (11.8 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.55 g (1.9 mmol, 32.2%) of **19e** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

3-[10-(1,2,4-Trioxolan-3-yl)decyl]-1,2,4-trioxolane (19e): Colorless liquid. − ^{1}H NMR: δ = 1.27−1.43 (m, 16 H), 1.70 (m, 4 H), 5.02 (s, 2 H), 5.13 (t, J = 5.12 Hz, 2 H), 5.18 (s, 2 H). − ^{13}C NMR: δ = 24.18, 29.71, 31.46, 94.33, 104.16. − $C_{14}H_{26}O_6$ (290.3): calcd. C 57.87, H 8.96; found C 58.28, H 8.62. − Reduction of **19e** with TPP gave 1,12-dodecanedial.

Ozonolysis of 9a in the Presence of 6d: Ozonolysis of 0.54 g (8.0 mmol) of **9a** and 1.05 g (24.0 mmol) of **6d** in 50 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.48 g (3.0 mmol, 37.4%) of **17f** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

(5-Methyl-1,2,4-trioxolan-3-yl)butanal (17f): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.38 (d, J = 8.23 Hz, 3 H), 1.74−1.78 (m, 4 H), 2.49 (t, J = 6.15 Hz, 2 H), 5.18−5.32 (m, 2 H), 9.75 (t, J = 2.08 Hz, 1 H). − ^{13}C NMR: δ = 16.67, 18.14, 30.27, 32.43, 32.47, 43.68, 101.84, 104.10, 202.14. − $C_7H_{12}O_4$ (160.2): calcd. C 52.48, H 7.55; found C 52.58, H 7.61. − Reduction of **17f** with TPP gave 1,5-pentanedial [δ = 1.90 (quint), 2.47 (t), 9.72 (s)] and actaldehyde (**6d**) in a ratio of ca. 1:1.

Ozonolysis of Isopropenyl Acetate in the Presence of *cis*- and *trans*-17f: Ozonolysis of 0.64 g (4.0 mmol) of *cis*- and *trans*-**17f** and 0.8 g (8.0 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.17 g (0.82 mmol, 20.8%) of **19f** was isoated [solvent: dichloromethane/diethyl ether, 15:1].

5-Methyl-3-[3-(1,2,4-trioxolane-3-yl)propyl]-1,2,4-trioxolane (19f): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.42 (d, J = 6.12 Hz, 3 H), 1.57 (m, 2 H), 1.71 (m, 4 H), 5.04 (s, 1 H), 5.12−5.37 (m, 3 H), 5.15 (s, 1 H). − ^{13}C NMR: δ = 14.98, 16.63, 18.22, 18.33, 18.49, 29.81, 30.71, 31.21, 31.28, 32.82, 94.39, 101.60, 101.77, 101.88, 103.62, 104.14, 104.24. − $C_8H_{14}C_6$ (206.2): calcd. C 46.59, H 6.84; found C 46.72, H 6.68. − Reduction of **19f** with TPP gave 1,5-pentanedial [δ = 1.90 (quint), 2.47 (t), 9.72 (s)] and acetaldehyde (**6d**) in a ratio of ca. 1:1.

Ozonolysis of 9b in the Presence of 6d: Ozonolysis of 0.66 g (8.0 mmol) of **9b** and 1.05 g (24.0 mmol) of **6d** in 50 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.16 g (0.92 mmol, 10.1%) of **18g** and 0.38 g (2.16 mmol, 27.4%) of **17g** were isolated [solvent: dichloromethane/diethyl ether, 15:1].

9-Methyl-7,8,10,11-tetraoxabicyclo[4.4.1]undecane (18g): Colorless liquid. − ^{1}H NMR: δ = 1.30−1.48 (m, 4 H), 1.38 (d, J = 6.14 Hz, 3 H), 4.83 (m, 1 H). − ^{13}C NMR (BB): δ = 16.69, 18.51, 23.79, 30.89, 32.63, 34.47, 101.54, 101.66, 104.50. − ^{13}C NMR (CB): 100.36, 100.40, 101.75, 101.79, 103.21, 104.64. − $C_8H_{14}O_4$ (174.2): calcd. C 55.16, H 8.10; found C 54.88, H 7.97. − Reduction of **18g** with TPP gave 1,6-hexanedial [δ = 1.66 (t), 2.33 (t), 9.72 (s)] and acetaldehyde (**6d**) in a ratio of ca. 1:1.

(5-Methyl-1,2,4-trioxolan-3-yl)pentanal (17g): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.40 (d, J = 6.15 Hz, 3 H), 1.45−1.60 (m, 2 H), 1.65−1.72 (m, 4 H), 2.46 (t, J = 6.11 Hz, 2 H), 5.19−5.35 (m, 2 H), 9.77 (s, 1 H). − ^{13}C NMR: δ = 16.24, 17.91, 21.69, 23.40, 30.42, 32.47, 43.51, 101.15, 101.30, 103.85, 103.95, 202.06. − $C_8H_{14}O_4$ (174.2): calcd. C 55.16, H 8.10; found C 54.88, H 7.97. − Reduction of **17g** with TPP gave 1,6-hexanedial and acetaldehyde (**6d**) in a ratio of ca. 1:1.

Ozonolysis of Isopropenyl Acetate in the Presence *cis*- and *trans*-17g: Ozonolysis of 0.60 g (3.5 mmol) of *cis*- and *trans*-**17g** and 0.7 g (7.0 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.27 g (1.23 mmol, 35.1%) of *cis*- and *trans*-**19g** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

5-Methyl-3-[4-(1,2,4-trioxolan-3-yl)butyl]-1,2,4-trioxolane (19g): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.39−1.49 (m, 4 H), 1.44 (d, J = 6.08 Hz, 3 H), 1.62−1.77 (m, 4 H), 5.03 (s, 1 H), 5.11−5.35 (m, 3 H), 5.16 (s, 1 H). − ^{13}C NMR:

δ = 16.67, 18.41, 23.87, 23.99, 31.33, 31.35, 32.93, 94.40, 101.59, 101.74, 103.85, 103.87, 104.37, 104.46. − $C_9H_{16}O_6$ (220.2): calcd. C 49.18, H 7.32; found C 48.98, H 7.46. − Reduction of **19g** with TPP gave 1,6-hexanedial [δ = 1.66 (t), 2.33 (t), 9.72 (s)] and acetaldehyde (**6d**) in a ratio of ca. 1:1.

Ozonolysis of 9c in the Presence of 6d: Ozonolysis of 0.77 g (8.0 mmol) of **9c** and 1.05 g (24.0 mmol) of **6d** in 50 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.18 g (0.76 mmol, 8.4%) of **18h** and 0.27 g (0.90 mmol, 17.3%) of **17h** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

10-Methyl-8,9,11,12-tetraoxabicyclo[5.4.1]dodecane (18h): Colorless liquid. − ^{1}H NMR: δ = 1.32−1.49 (m, 6 H), 1.37 (d, *J* = 6.12 Hz, 3 H), 1.58−1.72 (m, 4 H), 4.83 (m, 1 H), 5.14−5.35 (m, 2 H). − ^{13}C NMR: δ = 16.61, 17.43, 18.48, 23.71, 30.84, 32.33, 34.42, 101.54, 101.66, 104.50. − $C_9H_{16}O_4$ (188.2): calcd. C 57.44, H 8.57; found C 57.51, H 8.43. − Reduction of **18h** with TPP gave 1,7-heptanedial [δ = 1.36 (quint), 1.63 (quint), 2.45 (t), 9.71 (s)] and acetaldehyde (**6d**) in a ratio of ca. 1:1.

(5-Methyl-1,2,4-trioxolan-3-yl)hexanal (17h): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.35−1.46 (m, 4 H), 1.40 (d, *J* = 6.10 Hz, 3 H), 1.62−1.71 (m, 4 H), 2.44 (t, *J* = 5.12 Hz, 2 H), 5.16−5.21 (m, 1 H), [5.26 (q, *J* = 5.12 Hz), 5.33 (q, *J* = 5.12 Hz)] (1 H), 9.75 (s, 1 H). − ^{13}C NMR: δ = 16.69, 18.48, 20.97, 22.18, 23.86, 28.96, 30.85, 32.86, 44.04, 101.58, 101.72, 104.49, 104.57, 202.91. − $C_9H_{16}O_4$ (188.2): calcd. C 57.44, H 8.57; found C 57.62, H 8.38. − Reduction of **17h** with TPP gave 1,7-heptanedial [δ = 1.36 (quint), 1.63 (quint), 2.45 (t), 9.71 (s)] and acetaldehyde (**6d**) in a ratio of ca. 1:1.

Ozonolysis of Isopropenyl Acetate in the Presence of *cis*- and *trans*-17h: Ozonolysis of 0.50 g (2.7 mmol) of *cis*- and *trans*-**17h** and 0.55 g (5.5 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.06 g (0.26 mmol, 9.6%) of *cis*- and *trans*-**19h** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

5-Methyl-3-[5-(1,2,4-trioxolan-3-yl)pentyl]-1,2,4-trioxolane (19h): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.31−1.46 (m, 6 H), 1.38 (d, *J* = 6.23 Hz, 3 H), 1.66−1.73 (m, 4 H), 5.02 (s, 1 H), 5.12 (t, *J* = 5.12 Hz, 1 H), 5.18 (s, 1 H), 5.19 (m, 1 H), [5.26 (q, *J* = 5.12 Hz), 5.33 (q, *J* = 5.12 Hz)] (1 H). − ^{13}C NMR: δ = 16.25, 18.04, 20.53, 23.19, 23.54, 23.56, 28.94, 29.07, 30.48, 32.44, 93.94, 98.43, 101.20, 103.59, 104.11. − $C_{10}H_{18}O_6$ (234.3): calcd. C 51.27, H 7.74; found C 51.43, H 7.62. − Reduction of **19h** with TPP gave 1,7-heptanedial [δ = 1.36 (quint), 1.63 (quint), 2.45 (t), 9.71 (s)] and acetaldehyde (**6d**) in a ratio of ca. 1:1.

Ozonolysis of 9d in the Presence of 6d: Ozonolysis of 0.50 g (4.54 mmol) of **9d** and 1.20 g (27.0 mmol) of **6d** in 50 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.07 g (0.35 mmol, 7.7%) of **18i** and 0.18 g (0.89 mmol, 19.2%) of **17i** were isolated [solvent: dichloromethane/diethyl ether, 15:1].

11-Methyl-9,10,12,13-tetraoxabicyclo[6.4.1]tridecane (18i): Colorless liquid. − ^{1}H NMR: δ = 1.34−1.43 (m, 8 H), 1.39 (d, *J* = 6.23 Hz, 3 H), 1.62−1.69 (m, 4 H), 4.84 (m, 3 H). − ^{13}C NMR (BB): δ = 16.29, 18.18, 23.31, 29.11, 30.67, 32.57, 34.29, 101.14, 101.24. 101.52, 104.58. − ^{13}C NMR (CB): δ = 100.52, 100.87, 101.95, 102.14, 103.58, 104.96. − $C_{10}H_{18}O_4$ (202.2): calcd. C 59.40, H 8.97; found C 59.30, H 8.93. − Reduction of **18i** with TPP gave 1,8-octanedial [δ = 1.36 (quint), 1.63 (quint), 2.45 (t), 9.72 (s)] and acetaldehyde (**4B**) in a ratio of ca. 1:1.

(5-Methyl-1,2,4-trioxolan-3-yl)heptanal (17i): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.35−1.42 (m, 6 H), 1.38 (d, *J* = 6.24 Hz, 3 H), 1.58−1.74 (m, 4 H), 2.42 (t, *J* = 6.23 Hz, 2 H), 5.16−5.37 (m, 2 H), 9.76 (s, 1 H). − ^{13}C NMR: δ = 16.65, 18.50, 23.20, 23.99, 29.57, 30.96, 32.92, 44.13, 101.64, 104.55, 203.05. − $C_{10}H_{18}O_4$ (202.2): calcd. C 59.40, H 8.97; found C 58.99, H 8.89. − Reduction of **17i** with TPP gave 1,8-octanedial and acetaldehyde (**6d**) in a ratio of ca. 1:1.

Ozonolysis of Isopropenyl Acetate in the Presence of *cis*- and *trans*-17i: Ozonolysis of 0.72 g (3.6 mmol) of **17i** and 0.72 g (7.2 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.08 g (0.32 mmol, 8.9%) of *cis*- and *trans*-**19i** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

5-Methyl-3-[6-(1,2,4-trioxolan-3-yl)hexyl]-1,2,4-trioxolane (19i): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.31−1.46 (m, 8 H), 1.37 (d, *J* = 6.23 Hz, 3 H), 1.65−1.75 (m, 4 H), 5.02 (s, 1 H), 5.12 (t, *J* = 5.12 Hz, 1 H), 5.17 (s, 1 H), 5.19 (m, 1 H), [5.24 (q, *J* = 5.12 Hz), 5.33 (q, *J* = 5.13 Hz)] (1 H). − ^{13}C NMR: δ = 16.62, 18.51, 23.99, 29.50, 30.97, 32.61, 32.94, 34.65, 94.31, 101.60, 104.54, 104.62. − $C_{11}H_{20}O_6$ (248.3): calcd. C 53.21, H 8.12; found C 53.19, H 8.07. − Reduction of **19i** with TPP gave 1,8-octanedial and acetaldehyde (**6d**) in a ratio of ca. 1:1.

Ozonolysis of 9e in the Presence of 6d: Ozonolysis of 1.33 g (8.0 mmol) of **9e** and 1.05 g (24.0 mmol) of **6d** in 50 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.36 g (1.4 mmol, 17.4%) *cis*- and *trans*-**17j** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

(5-Methyl-1,2,4-trioxolan-3-yl)undecane (17j): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.29−1.58 (m, 14 H), 1.38 (d, *J* = 6.23 Hz, 3 H), 1.60−1.74 (m, 4 H), 2.42 (t, *J* = 5.12 Hz, 2 H), 5.15−5.21 (m, 1 H), [5.27 (q, *J* = 5.12 Hz), 5.34 (q, *J* = 5.12 Hz)] (1 H), 9.76 (s, 1 H). − ^{13}C NMR: δ = 16.62, 18.56, 22.37, 24.07, 24.20, 29.65, 31.04, 32.96, 44.19, 101.55, 104.62, 203.10. − $C_{14}H_{26}O_4$ (258.3): calcd. C 65.08, H 10.14; found C 64.96, H 10.03. − Reduction of **17j** with TPP gave 1,12-dodecanedial [δ = 1.26−136 (m), 1.63 (quint), 2.45 (t), 9.71 (s)] and acetaldehyde (**6d**) in a ratio of ca. 1:1.

Ozonolysis of Isopropenyl Acetate in the Presence of *cis*- and *trans*-17j: Ozonolysis of 0.78 g (2.9 mmol) of **17j** and 0.60 g (6.0 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.09 g (0.29 mmol, 10.1%) of *cis*- and *trans*-**19j** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

5-Methyl-3-[10-(1,2,4-trioxolan-3-yl)decyl]-1,2,4-trioxolane (19j): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.31−1.46 (m, 16 H), 1.35 (d, *J* = 6.23 Hz, 3 H), 1.66−1.73 (m, 4 H), 5.00 (s, 1 H), 5.15 (m, 2 H), 5.22 (s, 1 H), 5.27 (m, 1 H). − ^{13}C NMR: δ = 16.63, 18.56, 24.16, 29.70, 31.09, 31.46, 32.97, 94.30, 101.46, 101.56, 104.13, 104.66, 104.74. − $C_{15}H_{28}O_6$ (304.4): calcd. C 59.19, H 9.27; found C 59.15, H 9.24. − Reduction of **19j** with TPP gave 1,12-dodecanedial and acetaldehyde (**6d**) in a ratio of ca. 1:1.

Ozonolysis of 9a in the Presence of 6b: Ozonolysis of 0.82 g (12.0 mmol) of **9a** and 1.66 g (24.0 mmol) of **6b** in 50 mL of dichloromethane at −78 °C gave a liquid residue, from which 1.03 g (5.56 mmol, 46.8%) of *cis*- and *trans*-**17k** was isolated [solvent: dichloromethane/diethyl ether, 15:1]. Additional separation of 0.60 g (3.2 mmol) of this mixture led to 0.24 g (1.3 mmol, 40.6%) of *trans*-**17k**.

(5-Cyano-5-methyl-1,2,4-trioxolan-3-yl)butanal (17k): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.60−1.80 (m, 4 H), 1.74 (s, 3 H), 2.24 (t, *J* = 6.23 Hz, 2 H), [5.14 (t, *J* = 5.12 Hz), 5.52 (t, *J* = 5.12 Hz)] (1 H), 9.63 (t, *J* = 2.33 Hz, 1 H). − ^{13}C NMR: δ = 15.58, 15.79, 20.59, 20.61, 28.58, 32.67, 42.57, 42.73, 97.73, 98.15, 105.79, 115.79, 116.40, 201.01. − $C_8H_{11}NO_4$ (185.2): calcd. C 51.90, H 6.00; found C 51.74, H 5.96. − ***trans*-17k:** Colorless liquid. − ^{1}H NMR: δ = 1.82−1.93 (m, 4 H), 1.85 (s, 3 H), 2.56 (t, *J* = 6.23 Hz, 2 H), 5.26 (t, *J* = 5.12 Hz, 1 H), 9.78 (t, *J* = 1.88 Hz, 1 H). − ^{13}C NMR: δ = 15.74, 20.81, 28.79, 42.95, 98.29, 106.00, 116.51, 201.00. − Reduction of **17k** with TPP gave 1,5-pentanedial [δ = 1.90 (quint), 2.47 (t), 9.72 (s)] and acetyl cyanide (**6b**) in a ratio of ca. 1:1.

Ozonolysis of Isopropenyl Acetate in the Presence of *cis*- and *trans*-17k: Ozonolysis of 0.30 g (1.6 mmol) of *trans*-**17k** and 0.30 g (3.0 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.07 g (0.3 mmol, 18.8%) of *trans*-**19k** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

5-Cyano-5-methyl-3-[3-(1,2,4-trioxolan-3-yl)propyl]-1,2,4-trioxolane (*trans*-19k): Colorless liquid. − ^{1}H NMR: δ = 1.66−1.95 (m, 6 H), 1.85 (s, 3 H), 5.06 (s, 1 H), 5.17 (m, 1 H), 5.18 (s, 1 H), 5.25 (t, *J* = 5.12 Hz, 1 H). − ^{13}C NMR: δ = 17.53, 20.80, 29.37, 30.71, 93.99, 98.26, 102.98, 106.04, 116.52. − $C_9H_{13}NO_6$ (231.2): calcd. C 46.76, H 5.67; found C 46.42, H 5.51. − Reduction of **18k** with TPP gave 1,5-pentanedial and acetyl cyanide (**6b**) in a ratio of ca. 1:1.

Ozonolysis of 9b in the Presence of 6b: Ozonolysis of 0.98 g (12.0 mmol) of **9b** and 1.65 g (24.0 mmol) of **6b** in 50 mL of dichloromethane at −78 °C gave a liquid residue, from which 1.67 g (8.4 mmol, 70.1%) of *cis*- and *trans*-**17l** was isolated [solvent: dichloromethane/diethyl ether, 15:1]. Additional separation of 0.83 g (4.2 mmol) of this mixture led to 0.36 g (1.8 mmol, 43.4%) of *trans*-**17l**.

(5-Cyano-5-methyl-1,2,4-trioxolan-3-yl)pentanal (17l): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.37−1.84 (m, 6 H), 1.79 (s, 3 H), 2.42 (t, *J* = 6.23 Hz, 2 H), [5.19 (t, *J* = 5.12 Hz), 5.55 (t, *J* = 5.12 Hz)] (1 H), 9.70 (t, *J* = 1.59 Hz, 1 H). − ^{13}C NMR: δ = 20.72, 20.81, 21.30, 21.52, 22.84, 23.03, 29.27, 33.32, 43.27, 97.85, 98.27, 106.19, 116.0, 116.63, 202.14. − $C_9H_{13}NO_4$ (199.2): calcd. C 54.27, H 6.58; found C 54.64, H 6.49. − ***trans*-17l:** Colorless liquid. − ^{1}H NMR: δ = 1.53−1.56 (m, 2 H), 1.69−1.71 (m, 2 H), 1.83−1.90 (m, 2 H), 1.85 (s, 3 H), 2.46 (t, *J* = 6.23 Hz, 2 H), 5.26 (t, *J* = 5.12 Hz, 1 H), 9.77 (t, *J* = 1.58 Hz, 1 H) − ^{13}C NMR: δ = 20.63, 21.37, 22.57, 29.17, 43.19, 98.08, 106.00, 116.47, 201.74. − Reduction of **17l** with TPP gave 1,6-hexanedial [δ = 1.66 (t), 2.33 (t), 9.72 (s)] and acetyl cyanide (**6b**) in a ratio of ca. 1:1.

Ozonolysis of Isopropenyl Acetate in the Presence of *trans*-17l: Ozonolysis of 0.38 g (1.9 mmol) of *trans*-**17l** and 0.38 g (3.8 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.15 g (0.61 mmol, 32.1%) of *trans*-**19l** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

5-Cyano-5-methyl-3-[4-(1,2,4-trioxolan-3-yl)butyl]-1,2,4-trioxolane (*trans*-19l): Colorless liquid. − ^{1}H NMR: δ = 1.46−1.88 (m, 8 H), 1.83 (s, 3 H), 5.02 (s, 1 H), 5.12−5.16 (m, 1 H), 5.14 (s, 1 H), 5.25 (t, *J* = 5.12 Hz, 1 H). − ^{13}C NMR: δ = 20.92, 23.11, 23.44, 29.51, 30.87, 94.02, 98.26, 103.35, 106.28, 116.62. − $C_{10}H_{15}NO_6$ (245.2): calcd. C 48.98, H 6.17; found C 49.27, H 6.11. − Reduction of **19l** with TPP gave 1,6-hexanedial and acetyl cyanide (**6b**) in a ratio of ca. 1:1.

Ozonolysis of 9c in the Presence of 6b: Ozonolysis of 0.29 g (3.0 mmol) of **9c** and 0.41 g (6.0 mmol) of **6b** in 50 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.39 g (1.83 mmol, 61.0%) of *cis*- and *trans*-**17m** was isolated [solvent: dichloromethane/diethyl ether, 15:1]. Additional separation of 0.42 g (2.0 mmol) of this mixture led to 0.12 g (0.56 mmol, 28.0%) of *trans*-**17m**.

(5-Cyano-5-methyl-1,2,4-trioxolan-3-yl)hexanal (17m): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.36−1.85 (m, 8 H), 1.84 (s, 3 H), 2.42 (t, *J* = 6.23 Hz, 2 H), [5.21 (t, *J* = 5.12 Hz), 5.58 (t, *J* = 5.12 Hz)] (1 H), 9.79 (s, 1 H). − ^{13}C NMR: δ = 20.81, 20.97, 21.59, 22.90, 23.20, 28.32, 28.53, 29.33, 33.26, 43.44, 97.80, 98.17, 106.20, 115.99, 116.63, 202.14. − $C_{10}H_{15}NO_4$ (213.2): calcd. C 56.33, H 7.09; found C 56.43, H 6.97. − ***trans*-17m:** Colorless liquid. − ^{1}H NMR: δ = 1.41−1.89 (m, 8 H), 1.85 (s, 3 H), 2.45 (t, *J* = 6.23 Hz, 2 H), 5.25 (t, *J* = 5.12 Hz, 1 H), 9.76 (t, *J* = 1.58 Hz, 1 H). − ^{13}C NMR: δ = 20.81, 21.56, 22.94, 28.61, 29.32, 43.41, 98.16, 106.29, 116.57, 202.12. − Reduction of **17m** with TPP gave 1,7-heptanedial [δ = 1.36 (quint), 1.63 (quint), 2.45 (t), 9.71 (s)] and acetyl cyanide (**6b**) in a ratio of ca. 1:1.

Ozonolysis of Isopropenyl Acetate in the Presence of *trans*-17m: Ozonoylsis of 0.23 g (1.1 mmol) of *trans*-**17m** and 0.23 g (2.3 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.15 g (0.58 mmol, 52.7%) of *trans*-**19m** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

5-Cyano-5-methyl-3-[5-(1,2,4-trioxolan-3-yl)pentyl]-1,2,4-trioxolane (*trans*-19m): Colorless liquid. − ^{1}H NMR: δ = 1.42−1.88 (m, 10 H), 1.85 (s, 3 H), 5.04 (s, 1 H), 5.14 (t, *J* = 5.12 Hz, 1 H), 5.18 (s, 1 H), 5.24 (t, *J* = 5.12 Hz, 1 H). − ^{13}C NMR: δ = 20.95, 23.16, 23.46, 28.99, 29.55, 30.91, 94.01, 98.23, 103.59, 106.47, 116.66. − $C_{11}H_{17}NO_6$ (259.3): calcd. C 50.96, H 6.61; found C 50.87, H 6.46. − Reduction of **19m** with TPP gave 1,7-heptanedial and acetyl cyanide (**6b**) in a ratio of ca. 1:1.

Ozonolysis of 9a in the Presence of 6c: Ozonolysis of 0.54 g (8.0 mmol) of **9a** and 2.09 g (16.0 mmol) of **6c** in 50 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.83 g (3.4 mmol, 42.3%) of *cis*- and *trans*-**17n** was isolated [solvent: dichloromethane/pentane, 15:1]. Additional separation of 1.95 g (7.9 mmol) of this mixture led to 0.73 g (2.9 mmol, 36.7%) of *trans*-**17n** [solvent: dichloromethane/*n*-pentane, 4:1].

(5-Cyano-5-phenyl-1,2,4-trioxolan-3-yl)butanal (17n): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.84−2.05 (m, 4 H), 2.42−2.64 (m, 2 H), [5.54 (t, *J* = 5.12 Hz), 5.85 (t, *J* = 5.12 Hz)] (1 H), 7.45−7.72 (m, 5 H), 9.79 (t, *J* = 2.08 Hz, 1 H). − ^{13}C NMR: δ = 15.95, 16.36, 29.10, 33.03, 42.97, 43.21, 100.93, 101.14, 106.95, 107.54, 116.05, 127.13, 132.27, 201.23. − $C_{13}H_{13}NO_4$ (247.3): calcd. C 63.15, H 5.30; found C 62.83, H 5.21. − ***trans*-17n:** Colorless liquid. − ^{1}H NMR: δ = 1.96 (m, 2 H), 2.05 (m, 2 H), 2.60 (t, *J* = 5.12 Hz, 2 H), 5.54 (t, *J* = 5.12 Hz), 7.45−7.72 (m, 5 H), 9.80 (s, 1 H). − ^{13}C NMR: δ = 15.95, 29.10, 43.21, 100.84, 107.49, 116.00, 127.10, 129.16, 132.22, 201.23. − Reduction of **17n** with TPP gave 1,5-pentanedial [δ = 1.90 (quint), 2.47 (t), 9.72 (s)] and benzoyl cyanide (**6c**) in a ratio of ca. 1:1.

Ozonolysis of Isopropenyl Acetate in the Presence of *trans*-17n: Ozonolysis of 0.73 g (2.9 mmol) of *trans*-**17n** and 0.57 g (5.7 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.21 g (0.72 mmol, 24.8%) of *trans*-**19n** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

5-Cyano-5-phenyl-3-[3-(1,2,4-trioxolan-3-yl)propyl]-1,2,4-trioxolane (*trans*-19n): Colorless liquid. − ^{1}H NMR: δ = 1.68 (m, 2 H), 1.77 (m, 2 H), 1.97 (m, 2 H), 4.98 (s, 1 H), 5.09 (s, 1 H), 5.12 (t, *J* = 5.12 Hz, 1 H), 5.45 (t, *J* = 5.12 Hz, 1 H), 7.40−7.63 (m, 5 H). − ^{13}C NMR: δ = 17.62, 29.51, 30.80, 94.05, 103.03, 107.43, 115.90, 127.02, 128.84, 129.05, 134.07. − $C_{14}H_{15}NO_6$ (293.3): calcd. C 57.33, H 5.16; found C 57.21, H 5.02. − Reduction of **19n** with TPP gave 1,5-pentanedial and benzoyl cyanide (**6c**) in a ratio of ca. 1:1.

Ozonolysis of 9b in the Presence of 6c: Ozonolysis of 0.32 g (4.7 mmol) of **9b** and 1.04 g (8.0 mmol) of **6c** in dichloromethane at −78 °C gave a liquid residue, from which 0.75 g (2.9 mmol, 61.7%) of *cis*- and *trans*-**17o** was isolated [solvent: dichloromethane/pentane, 15:1]. Additional separation of 1.95 g (7.3 mmol) of this mixture led to 0.50 g (1.91 mmol, 26.2%) of *trans*-**17o** [solvent: dichloromethane/*n*-pentane, 10:1].

(5-Cyano-5-phenyl-1,2,4-trioxolan-3-yl)pentanal (17o): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.15−2.05 (m, 6 H), 2.44−2.53 (m, 2 H), [5.53 (t, *J* = 5.12 Hz), 5.82 (t, *J* = 5.12 Hz)] (1 H), 7.44−7.72 (m, 5 H), 9.77 (t, *J* = 2.06 Hz, 1 H). − ^{13}C NMR: δ = 21.41, 21.66, 22.83, 23.24, 29.61, 33.42, 43.43, 100.56, 100.98, 107.06, 107.64, 115.52, 116.03, 126.10−136.88 (m), 201.94. − $C_{14}H_{15}NO_4$ (261.4): calcd. C 64.33, H 5.83; found C 64.42, H 5.77. − ***trans*-17o:** Colorless liquid. − ^{1}H NMR: δ = 1.68 (m, 4 H), 1.94−2.04 (m, 2 H), 2.48 (t, *J* = 6.23 Hz, 2 H), 5.53 (t, *J* = 5.12 Hz, 1 H), 7.42−7.73 (m, 5 H), 9.75 (t, *J* = 2.08 Hz, 1 H). − ^{13}C NMR: δ = 22.04, 23.22, 29.28, 43.79, 101.38, 107.83, 116.07, 127.45, 129.21, 129.53, 132.57, 202.37. − Reduction of **17o** with TPP gave 1,6-hexanedial [δ = 1.66 (t), 2.33 (t), 9.72 (s)] and benzoyl cyanide (**6c**) in a ratio of ca. 1:1.

Ozonolysis of Isopropenyl Acetate in the Presence of *trans*-17o: Ozonolysis of 1.04 g (4.0 mmol) of *trans*-**17o** and 0.80 g (8.0 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.32 g (1.04 mmol, 26.2%) of *trans*-**19o** was isolated [solvent: dichloromethane/*n*-pentane, 2:1].

5-Cyano-5-phenyl-3-[4-(1,2,4-trioxolan-3-yl)butyl]-1,2,4-trioxolane (*trans*-19o): Colorless liquid. − ^{1}H NMR: δ = 1.48−1.56 (m, 4 H), 1.66−1.72 (m, 2 H), 1.91−1.94 (m, 2 H), 4.97 (s, 1 H), 5.07 (t, *J* = 5.12 Hz, 1 H), 5.09 (s, 1 H), 5.44 (t, *J* = 5.12 Hz, 1 H), 7.38−7.63 (m, 5 H). − ^{13}C NMR: δ = 23.39, 23.75, 29.98, 31.16, 94.29, 101.22, 103.63, 107.93, 116.25, 127.32, 129.23, 129.34, 132.33. − $C_{15}H_{17}NO_6$ (307.3): calcd. C 58.63, H 5.58; found C 58.73, H 5.64. − Reduction of **19o** with TPP gave 1,6-hexanedial and benzoyl cyanide (**6c**) in a ratio of ca. 1:1.

Ozonolysis of 9c in the Presence of 6c: Ozonolysis of 0.77 g (8.0 mmol) of **9c** and 2.09 g (16.0 mmol) of **6c** in 50 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.72 g (2.6 mmol, 32.7%) of *cis*- and *trans*-**17p** was isolated [solvent: dichloromethane/*n*-pentane, 5:1]. Additional separation of 2.14 g (7.8 mmol) of this mixture led to 0.62 g (2.2 mmol, 28.4%) of *trans*-**17p**.

(5-Cyano-5-phenyl-1,2,4-trioxolan-3-yl)hexanal (17p): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.42−2.01 (m, 8 H), 2.39−2.49 (m, 2 H), [5.52 (t, *J* = 5.12 Hz), 5.81 (t, *J* = 5.12 Hz)] (1 H), 7.43−7.73 (m, 5 H), 9.76 (t, *J* = 2.08 Hz, 1 H). − ^{13}C NMR: δ = 21.71, 23.11, 23.28, 28.56, 28.80, 29.81, 33.38, 43.56, 100.99, 101.38, 107.26, 107.83, 116.03, 116.07, 127.01−132.12, 202.30. − $C_{15}H_{17}NO_4$ (275.3): calcd. C 65.44, H 6.23; found C 65.37, H 6.11. − ***trans*-17p:** Colorless liquid. − ^{1}H NMR: δ = 1.38 (m, 2 H), 1.56 (m, 4 H), 1.92 (m, 2 H), 2.44 (t, *J* = 5.12 Hz, 2 H), 5.50 (t, *J* = 5.12 Hz, 1 H), 7.45−7.70 (m, 5 H), 9.75 (t, *J* = 2.08 Hz, 1 H). − ^{13}C NMR: δ = 21.59, 22.98, 28.67, 29.54, 43.56, 100.99, 107.83, 115.93, 127.01−132.12(m), 202.30. − Reduction of **17p** with TPP gave 1,7-heptanedial [δ = 1.36 (quint), 1.63 (quint), 2.45 (t), 9.71 (s)] and benzoyl cyanide (**6c**) in a ratio of ca. 1:1.

Ozonolysis of Isopropenyl Acetate in the Presence of *trans*-17p: Ozonolysis of 0.80 g (3.0 mmol) of *trans*-**17p** and 0.60 g (6.0 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.33 g (1.03 mmol, 34.3%) of *trans*-**19p** was isolated [solvent: dichloromethane/*n*-pentane, 4:1].

5-Cyano-5-phenyl-3-[5-(1,2,4-trioxolan-3-yl)pentyl]-1,2,4-trioxolane (*trans*-19p): Colorless liquid. − ^{1}H NMR: δ = 1.38−1.62 (m, 6 H), 1.74 (m, 2 H), 2.00 (m, 2 H), 5.03 (s, 1 H), 5.10 (t, *J* = 5.12 Hz, 1 H), 5.13 (s, 1 H), 5.52 (t, *J* = 5.12 Hz, 1 H), 7.45−7.71 (m, 5 H). − ^{13}C NMR: δ = 23.06, 23.37, 28.90, 29.60, 30.81, 93.88, 100.86, 103.47, 107.79, 115.96, 126.90, 128.99, 131.94. − $C_{16}H_{19}NO_6$ (321.3): calcd. C 59.81, H 5.96; found C 59.42, H 5.83. − Reduction of **19p** with TPP gave 1,7-heptanedial and benzoyl cyanide (**6c**) in a ratio of ca. 1:1.

Ozonolysis of 10a in the Presence of 6a: Ozonolysis of 0.25 g (3 mmol) of **10a** and 1 mL of **6a** (freshly prepared by pyrolysis of paraformaldehyde) in 50 mL of dichloromethane at −78 °C, followed by distillation of the solvent under reduced pressure, gave a liquid residue, from which 0.29 g (2.21 mmol, 73.8%) of **22** was isolated [solvent: diethyl ether/dichloromethane, 1:15]. − From the ozonolysis of **10a** in the presence of **6b** and **6c**, 0.24 g (1.18 mmol, 62.5%) and 0.23 g (1.82 mmol, 60.6%) of **22** was isolated, respectively.

1-Methyl-6,7,8-trioxabicyclo[3.2.1]octane (22): Colorless liquid. − ^{1}H NMR ($CDCl_3$): δ = 1.43 (s, 3 H), 1.54−1.75 (m, 5 H), 2.08−2.15 (m, 1 H), 5.19 (s, 1 H). − ^{13}C NMR: δ = 16.16, 21.31, 29.26, 33.95, 103.16, 108.05. Ozonide **22** was identified on the basis of comparison of its ^{1}H-NMR and ^{13}C-NMR data with those reported in the literature.[8] − Reduction of **22** with TPP gave 5-oxohexanal [δ = 1.71 (quint, *J* = 4.64 Hz, 2 H), 1.96 (s, 1 H), 2.30 (t, *J* = 4.64 Hz, 4 H)].

Ozonolysis of 10b in the Presence of 6a: Ozonolysis of 0.29 g (3 mmol) **10b** and 1 mL of formaldehyde (**6a**) (freshly prepared by pyrolysis of paraformaldehyde) in 50 mL of dichloromethane at −78 °C, followed by distillation of the solvent under reduced pressure, gave a liquid residue, from which 0.26 g (1.49 mmol, 49.7%) of **20d** was isolated [solvent; diethyl ether/*n*-pentane, 1:4].

(3-Methyl-1,2,4-trioxolan-3-yl)hexanal (20d): Colorless liquid. − ^{1}H NMR ($CDCl_3$): δ = 1.43 (s, 3 H), 1.46 (m, 2 H), 1.67 (m, 4 H), 2.47 (m, 2 H), 5.06 (d, *J* = 6.23 Hz, 1 H), 5.13 (d, *J* = 6.46 Hz, 1 H), 9.77 (s, 1 H). − ^{13}C NMR ($CDCl_3$): δ = 22.29, 22.83, 23.70, 37.30, 43.96, 94.28, 109.83, 202.26. − $C_8H_{14}O_4$ (174.2): calcd. C 55.16, H 8.10; found C 55.47, H 8.16. − Reduction of **20d** with TPP gave 5-oxohexanal.

Ozonolysis of Isopropenyl Acetate in the Presence of 20d: Ozonolysis of 0.52 g (3.0 mmol) of **20d** and 0.60 g (6.0 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.25 g (1.13 mmol, 37.6%) of **21d** was isolated [solvent: dichloromethane/*n*-pentane, 4:1].

3-Methyl-3-[4-(1,2,4-trioxolan-3-yl)butyl]-1,2,4-trioxolane (21d): Colorless liquid. − ^{1}H NMR: δ = 1.42 (s, 4 H), 1.49 (m, 4 H), 1.73 (m, 3 H), 5.03 (s, 1 H), 5.06 (d, *J* = 6.12 Hz, 1 H), 5.12 (m, 2 H), 5.18 (s, 1 H). − ^{13}C NMR: δ = 22.90, 24.00, 24.20, 31.38, 37.43, 94.35, 94.39, 103.93, 109.99. − $C_9H_{16}NO_6$ (220.2): calcd. C 49.09, H 7.32; found C 49.23, H 7.45. − Reduction of **21d** with TPP gave 5-oxohexanal.

Ozonolysis of 10b in the Presence of 6b: Ozonolysis of 0.29 g (3 mmol) of **10b** and 0.41 g (6 mmol) of **6b** in 50 mL of dichloromethane at −78 °C, followed by distillation of the solvent under reduced pressure, gave a liquid residue from which 0.26 g

(1.23 mmol, 41.0%) of **20e** was isolated [solvent: diethyl ether/*n*-pentane, 1:4].

(5-Cyano-3,5-dimethyl-1,2,4-trioxolan-3-yl)hexanal (20e): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.44 (s, 3 H), 1.62−1.69 (m, 4 H), [1.82 (s), 1.84 (s)] (3 H), 1.91 (m, 2 H), 2.47 (m, 2 H), 9.77 (s, 1 H). − ^{13}C NMR: δ = 20.61−24.74, 35.61, 39.03, 43.87, 98.40, 98.68, 114.17, 114.24, 117.19, 117.26, 202.46. − $C_{10}H_{15}NO_4$ (213.2): calcd. C 56.33, H 7.09; found C 56.65, H 7.02. − Reduction of **20e** with TPP gave 6-oxoheptanal [δ = 1.73 (quint, *J* = 4 Hz, 4 H), 1.98 (s, 3 H), 2.30 (q, *J* = 4 Hz, 4 H), 9.56 (s, 1 H)] and acetyl cyanide (**6b**) in a ratio of 1:1.

Ozonolysis of Isopropenyl Acetate in the Presence of *cis*- and *trans*-20e: Ozonolysis of 0.64 g (3.0 mmol) of *cis*- and *trans*-**20e** and 0.60 g (6.0 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.25 g (0.96 mmol, 32.0%) of *cis*- and *trans*-**21e** was isolated [solvent: dichloromethane/*n*-pentane, 4:1].

5-Cyano-3,5-dimethyl-3-[4-(1,2,4-trioxolan-3-yl)butyl]-1,2,4-trioxolane (21e): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.45 (s, 3 H), 1.71−1.90 (m, 6 H), [1.82 (s), 1.84 (s)] (3 H), 2.17 (m, 2 H), 5.05 (s, 1 H), 5.19 (d, *J* = 6.12 Hz, 1 H), 5.24 (s, 1 H). − ^{13}C NMR: δ = 20.69−24.85 (m), 31.29, 32.86, 35.76, 94.43, 98.68, 103.84, 114.22, 117.26. − $C_{11}H_{17}NO_6$ (259.2): calcd. C 50.96, H 6.61; found C 50.61, H 6.54. − Reduction of **21e** with TPP gave 6-oxoheptanal and acetyl cyanide (**6b**) in a ratio of 1:1.

Ozonolysis of 11 in the Presence of 6a: Ozonolysis of 0.39 g (3 mmol) of **11** and 1 mL of **6a** (freshly prepared by pyrolysis of paraformaldehyde) in 50 mL of dichloromethane at −78 °C, followed by distillation of the solvent under reduced pressure, gave a liquid residue, from which 0.40 g (1.9 mmol, 63.0%) of **25a** was isolated [solvent: dichloromethane/diethyl ether, 7:1].

***o*-[2-(1,2,4-Trioxolan-3-yl)ethyl]benzaldehyde (25a):** Colorless liquid. − ^{1}H NMR: δ = 2.00−2.07 (m, 2 H), 3.15−3.20 (m, 2 H), 5.09 (s, 1 H), 5.18 (s, 1 H), 5.22 (t, *J* = 6.12 Hz, 1 H), 7.29−8.34 (m, 4 H), 10.20 (s, 1 H). − ^{13}C NMR: δ = 27.52, 33.37, 94.46, 103.28, 127.38, 131.52, 133.81, 134.10, 134.26, 143.55, 193.00. − $C_{11}H_{12}O_4$ (208.2): calcd. C 63.46, H 5.81; found C 63.73, H 5.69. − Reduction of **25a** with TPP gave 3-(*o*-formylphenyl)propanal [^{1}H NMR: δ = 2.72 (t, *J* = 6.45 Hz, 2 H), 3.29 (t, *J* = 6.45 Hz, 2 H), 7.40−8.03 (m, 4 H), 9.73 (s, 1 H), 10.12 (s, 1 H). − ^{13}C NMR: δ = 25.73, 45.16, 127.29−143.03, 193.06, 201.23].

Ozonolysis of 11 in the Presence of 6b: Ozonolysis of 0.39 g (3 mmol) of **11** and 0.63 g (9 mmol) of **6b** in 50 mL of dichloromethane at −78 °C, followed by distillation of the solvent under reduced pressure, gave a liquid residue, from which 0.59 g (2.4 mmol, 80%) of **25b** was isolated [solvent: dichloromethane/diethyl ether, 7:1]. Additional separation led to 0.41 g (1.65 mmol, 55.2%) of *trans*-**25b**.

***o*-[2-(5-Cyano-5-methyl-1,2,4-trioxolan-3-yl)ethyl]benzaldehyde (25b):** Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.87 (d, *J* = 6.23 Hz, 3 H), [1.97−2.02 (m), 2.13−2.20 (m)] (2 H), 3.10−3.24 (m, 2 H), [5.32 (t, *J* = 6.12 Hz), 5.70 (t, *J* = 6.12 Hz)] (1 H), 7.27−8.34 (m, 4 H), 10.15 (s, 1 H). − ^{13}C NMR: δ = 21.26, 27.32, 27.66, 31.46, 35.66, 98.73, 98.81, 106.17, 106.23, 116.56, 117.15, 127.6, 131.60, 134.08, 134.34, 134.72, 142.74, 193.32, 193.37. − $C_{13}H_{13}NO_4$ (247.3): calcd. C 63.14, H 5.31; found C 62.77, H 5.27. − ***trans*-25b:** ^{1}H NMR: δ = 1.80 (s, 3 H), 2.07−2.14 (m, 2 H), 3.12−3.18 (m, 2 H), 5.25 (t, *J* = 6.12 Hz, 1 H), 7.19−8.28 (m, 4 H), 10.09 (s, 1 H). − ^{13}C NMR: δ = 21.38, 27.46, 31.53, 98.77, 106.26, 117.09, 127.68, 131.67, 134.13, 134.37, 134.91, 141.66, 193.31. − Reduction of **25b** with TPP gave 3-(*o*-formylphenyl)propanal and acetyl cyanide (**6b**) in a ratio of ca. 1:1.

Ozonolysis of 11 in the Presence of 6c: Ozonolysis of 0.39 g (3 mmol) of **11** and 1.18 g (9 mmol) of **6c** in 50 mL of dichloromethane at −78 °C, followed by distillation of the solvent under reduced pressure, gave a liquid residue, from which 0.71 g (2.4 mmol, 78.1%) of **25c** was isolated [solvent: dichloromethane/diethyl ether, 7:1].

***o*-[2-(5-Cyano-5-phenyl-1,2,4-trioxolan-3-yl)ethyl]benzaldehyde (25c):** Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = [2.14−2.19 (m), 2.29−2.33 (m)] (2 H), 3.18−3.31 (m, 2 H), [5.59 (t, *J* = 6.12 Hz), 5.91 (t, *J* = 6.12 Hz)] (1 H), 7.44−7.73 (m, 4 H), [10.15 (s), 10.17 (s)] (1 H). − ^{13}C NMR: δ = 27.47, 27.95, 31.763, 35.54, 101.08, 101.49, 107.14, 107.66, 115.95, 116.46, 127.52−142.64, 193.26, 193.30. − $C_{18}H_{15}NO_4$ (309.3): calcd. C 69.90, H 4.89; found C 70.12, H 5.03. − Reduction of **25c** with TPP gave 3-(*o*-formylphenyl)propanal and benzoyl cyanide (**6c**) in a ratio of 1:1.

Ozonolysis of 11 in the Presence of 6d: Ozonolysis of 0.39 g (3 mmol) of **11** and 0.39 g (9 mmol) of acetaldehyde **6d** in 50 mL of dichloromethane at −78 °C, followed by distillation of the solvent under reduced pressure, gave a liquid residue, from which 0.49 g (2.2 mmol, 74.2%) of **25d** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

***o*-[2-(5-Methyl-1,2,4-trioxolane-3-yl)ethyl]benzaldehyde (25d):** Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = [1.43 (s), 1.44 (s)] (3 H), 1.98−2.08 (m, 2 H), 3.14−3.21 (m, 2 H), 5.24−5.36 (m, 2 H), 7.23−7.83 (m, 4 H), 10.23 (s, 1 H). − ^{13}C NMR: δ = 16.73, 17.98, 27.41, 32.82, 35.14, 101.63, 101.81, 103.76, 103.88, 127.32, 130.81, 131.42, 131.53, 134.17, 143.61, 143.76, 192.73, 192.77. − $C_{12}H_{14}O_4$ (222.2): calcd. C 64.87, H 6.35; found C 64.58, H 6.46. − Reduction of **25d** with TPP gave 3-(*o*-formylphenyl)propanal and acetaldehyde (**6d**) in a ratio of 1:1.

Ozonolysis of Isopropenyl Acetate in the Presence of 25a: Ozonolysis of 0.62 g (3.0 mmol) of **25a** and 0.60 g (6 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a solid residue, from which 0.43 g (1.7 mmol, 58.3%) of **26a** was isolated [solvent: diethyl ether/dichloromethane, 1:7].

3-[*o*-(1,2,4-Trioxolan-3-yl)benzylmethyl]-1,2,4-trioxolane (26a): Colorless liquid. − ^{1}H NMR: δ = 1.99−2.10 (m, 2 H), 2.85−2.90 (m, 2 H), 5.09 (s, 1 H), 5.16 (s, 1 H), 5.21 (t, *J* = 6.12 Hz, 1 H), 5.32 (s, 1 H), 5.45 (s, 1 H), 6.23 (s, 1 H), 7.22−8.33 (m, 4 H). − ^{13}C NMR: δ = 26.74, 33.60, 94.58, 95.52, 101.44, 103.14, 127.14, 128.09, 130.36, 130.39, 130.88, 140.97. − $C_{12}H_{14}O_6$ (254.2): calcd. C 56.70, H 5.55; found C 56.97, H 5.67. − Reduction of **26a** with TPP gave 3-(*o*-formylphenyl)propanal.

Ozonolysis of Isopropenyl Acetate in the Presence of 25b: Ozonolysis of 0.82 g (3.0 mmol) of **25b** and 0.60 g (6 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a solid residue, from which 0.67 g (2.3 mmol, 77.4%) of **26b** was isolated [solvent: diethyl ether/dichloromethane, 1:7]. Separation of the mixture by HPLC (3.2 × 25 cm LiChrosorb Si 60, solvent: dichloromethane/*n*-pentane, 15:1) gave 0.52 g (1.76 mmol, 59.1%) of *trans*-**26b**.

5-Cyano-5-methyl-3-[*o*-(1,2,4-trioxolan-3-yl)benzylmethyl]-1,2,4-trioxolane (26b): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.82 (s, 3 H), 1.95−2.19 (m, 2 H), 2.72−2.93 (m, 2 H), [5.25 (t, *J* = 3.78 Hz), 5.63(t, *J* = 3.78 Hz)] (1 H), 5.29 (s,1 H), 5.42 (s, 1 H), 6.21 (m, 1 H), 7.17−7.60 (m, 4 H). − ^{13}C NMR: δ = 21.30, 26.35, 26.69, 31.85, 36.05, 36.19, 95.53, 98.44, 98.91, 101.40, 101.48, 101.54, 105.92, 105.97, 116.52, 117.10, 127.33−140.30. − $C_{14}H_{15}NO_6$ (293.3): calcd. C 57.33, H 5.16; found C 57.56, H 5.24. − ***trans*-26b:** ^{1}H NMR: δ = 1.79 (s, 3 H), 2.07−2.16 (m, 2 H), 2.86 (t, J = 9 Hz, 2 H), 5.21 (t, J = 3.78 Hz, 1 H), 5.25 (s, 1 H), 5.38 (s, 1 H), 6.14 (d, J = 3.57 Hz, 1 H), 7.16−7.54 (m, 4 H). − ^{13}C NMR: δ = 21.36, 26.36, 31.87, 95.53, 98.86, 101.49, 105.94, 117.05, 127.38, 128.22, 128.27, 130.42, 130.98, 140.19. − Reduction of **26b** with TPP gave 3-(*o*-formylphenyl)propanal and acetyl cyanide (**6b**) in a ratio of 1:1.

Ozonolysis of Isopropenyl Acetate in the Presence of 25c: Ozonolysis of 0.92 g (3.0 mmol) of **25c** and 0.60 g (6 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a solid residue, from which 0.69 g (1.7 mmol, 55.3%) of **26c** was isolated [solvent: diethyl ether/dichloromethane, 1:7]. Separation of the mixture by HPLC (3.2 × 25 cm LiChrosorb Si 60, solvent: dichloromethane/*n*-pentane, 15:1) gave 0.41 g (1.14 mmol, 38.1%) of *trans*-**26c**.

5-Cyano-5-phenyl-3-[*o*-(1,2,4-trioxolan-3-yl)benzylmethyl]-1,2,4-trioxolane (26c): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = [2.13−2.20 (m), 2.27−2.32 (m)] (2 H), [2.88−2.98 (m), 3.02 (t, J = 3.89 Hz)] (2 H), 5.27 (s, 1 H), 5.40 (s, 1 H), [5.59 (t, J = 3.78 Hz), 5.91 (t, J = 3.78 Hz)] (1 H), [6.13 (d, J = 3.56 Hz), 6.25 (d, J = 3.56 Hz)] (1 H), 7.23−8.31 (m, 9 H). − ^{13}C NMR: δ = 26.40, 26.72, 32.08, 36.30, 95.58, 101.23, 101.46, 101.58, 101.68, 97.18, 106.97, 107.44, 116.68, 116.54, 127.43−140.30. − $C_{19}H_{17}NO_6$ (355.4): calcd. C 64.21, H 4.82; found C 63.96, H 4.78. − ***trans*-26c:** ^{1}H NMR: δ = 2.28−2.36 (m, 2 H), 3.03 (t, J = 3.89 Hz, 2 H), 5.33 (s, 1 H), 5.46 (s, 1 H), 5.56 (t, J = 3.78 Hz, 1 H), 6.25 (d, J = 3.56 Hz, 1 H), 7.28−8.15 (m, 9 H). − ^{13}C NMR: δ = 26.43, 32.12, 95.53, 101.61, 107.31, 116.39, 127.43−140.20. − Reduction of **26c** with TPP gave 3-(*o*-formylphenyl)propanal and benzoyl cyanide (**6c**) in a ratio of 1:1.

Ozonolysis of Isopropenyl Acetate in the Presence of 25d: Ozonolysis of 0.66 g (3.0 mmol) of **25d** and 0.60 g (6 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a solid residue, from which 0.52 g (2.1 mmol, 69.4%) of **26d** was isolated [solvent: diethyl ether/dichloromethane, 1:7]. Separation of the mixture by HPLC (3.2 × 25 cm LiChrosorb Si 60, solvent: dichloromethane/*n*-pentane, 15:1) gave 0.43 g (1.59 mmol, 53.1%) of *trans*-**26d**.

5-Methyl-3-[*o*-(1,2,4-trioxolan-3-yl)benzylmethyl)]-1,2,4-trioxolane (26d): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.41 (d, J = 3.56 Hz, 3 H), 1.95−2.06 (m, 2 H), 2.81−2.88 (m, 2 H), 5.20−5.33 (m, 2 H), 5.30 (s, 1 H), 5.39 (s, 1 H), 6.22 (s, 1 H), 7.18−8.27 (m, 4 H). − ^{13}C NMR: δ = 16.77, 18.04, 26.63, 26.82, 33.06, 35.36, 95.50, 101.41, 101.74, 101.99, 103.68, 103.78, 127.04, 127.11, 130.50, 130.69, 130.86, 141.22. − IR (film): ṽ = 3150−2900 cm^{-1}, 1800, 1750. − $C_{13}H_{16}O_6$ (268.3): calcd. C 58.20, H 6.01; found C 57.96, H 6.10. − ***trans*-26d:** ^{1}H NMR: δ = 1.45 (d, J = 3.56 Hz, 3 H), 2.01−2.10 (m, 2 H), 2.88 (t, J = 9 Hz, 2 H), 5.25 (t, J = 3.78 Hz, 1 H), 5.35 (q, J = 6.12 Hz, 2 H), 5.46 (s, 1 H), 6.24 (s, 1 H), 7.22−7.61 (m, 4 H). − ^{13}C NMR: δ = 16.78, 26.61, 33.03, 95.51, 101.43, 102.01, 103.66, 127.13, 128.06, 130.32, 130.37, 130.88, 141.05. − Reduction of **26d** with TPP gave 3-(*o*-formylphenyl)propanal and acetaldehyde (**6d**) in a ratio of 1:1.

Ozonolysis of 12 in the Presence of 6b: Ozonolysis of 0.59 g (6.4 mmol) of **12** and 0.82 g (12.0 mmol) of **6b** in 50 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.23 g (1.1 mmol, 18.0%) of *cis*- and *trans*-**27b** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

(5-Cyano-5-methyl-1,2,4-trioxolan-3-yl)-3-cyclopentanecarbaldehyde (27b): Colorless liquid. − ^{1}H NMR: δ = 1.51−2.30 (m, 6 H), 1.86 (s, 3 H), 2.52 (m, 1 H), 2.85 (m, 1 H), 5.16−5.48 (m, 1 H), 9.65 (m, 1 H). − ^{13}C NMR: δ = 20.62, 20.80, 25.91, 26.00, 26.51, 26.70, 26.93, 27.16, 39.43, 42.91, 50.90, 51.05, 98.91, 98.95, 108.11, 116.42, 115.48, 202.19. − $C_{10}H_{13}NO_4$ (211.2): calcd. C 56.87, H 6.20; found C 56.62, H 6.13. − Reduction of **27b** with TPP gave cyclopentanedicarbaldehyde [δ = 1.86−2.26 (m), 2.83 (m)] and acetyl cyanide (**6b**) in a ratio of ca. 1:1.

Ozonolysis of Isopropenyl Acetate in the Presence of *cis*- and *trans*-27b: Ozonolysis of 0.78 g (3.7 mmol) of *cis*- and *trans*-**27b** and 0.74 g (7.4 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.24 g (0.93 mmol, 25.1%) of *cis*- and *trans*-**28b** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

5-Cyano-5-methyl-3-[3-(1,2,4-trioxolan-3-yl)cyclopentyl]-1,2,4-trioxolane (28b): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.40−2.30 (m, 8 H), 1.86 (s, 3 H), 5.04−5.06 (m, 1 H), 5.07 (s, 1 H), 5.20 (s, 1 H), [5.16 (t, J = 4.67 Hz), 5.48 (t, J = 4.67 Hz)] (1 H). − ^{13}C NMR: δ = 20.78, 20.96, 26.54, 26.72, 28.42, 28.95, 29.13, 39.20, 40.52, 42.80, 94.30, 98.02, 98.44, 105.40, 108.44, 115.90, 116.57. − $C_{11}H_{15}NO_6$ (257.2): calcd. C 51.37, H 5.88; found C 51.74, H 5.95. − Reduction of **28b** with TPP gave cyclopentanedicarbaldehyde and acetyl cyanide (**6b**) in a ratio of ca. 1:1.

Ozonolysis of 12 in the Presence of 6c: Ozonolysis of 0.55 g (6.0 mmol) of **12** and 1.60 g (12.0 mmol) of **6c** in 50 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.46 g (1.68 mmol, 28.0%) of *cis*- and *trans*-**27c** was isolated [solvent: dichloromethane/*n*-pentane, 4:1]. Additional separation of 1.44 g (5.3 mmol) of this mixture led to 0.68 g (2.5 mmol, 47.2%) of *trans*-**27c**.

(5-Cyano-5-phenyl-1,2,4-trioxolan-3-yl)-3-cyclopentanecarbaldehyde (27c): Colorless liquid (a mixture of *cis* and *trans* isomers). ^{1}H NMR: δ = 1.60−2.22 (m, 6 H), 2.56 (m, 1 H), 2.86 (m, 1 H), [5.45 (d, J = 6.10 Hz), 5.66 (d, J = 6.10 Hz)] (1 H), 7.47−7.72 (m, 5 H), 9.66 (d, J = 4.04 Hz, 1 H). − ^{13}C NMR: δ = 26.42, 26.52, 27.41, 27.47, 40.12, 43.46, 51.48, 51.61, 101.62, 101.65, 109.78, 110.13, 115.89, 116.38, 127.36−137.34, 203.07. − $C_{15}H_{15}NO_4$ (273.3): calcd. C 65.92, H 5.53; found C 65.63, H 5.48. − ***trans*-27c:** Colorless liquid. − ^{1}H NMR: δ = 1.56−2.18 (m, 6 H), 2.54 (m, 1 H), 2.87 (m, 1 H), 5.43 (d, J = 6.10 Hz, 1 H), 7.44−7.70 (m, 5 H), 9.64 (d, J = 4.03 Hz, 1 H). − ^{13}C NMR: δ = 26.36, 27.38, 27.46, 40.05, 51.46, 101.47, 110.10, 116.22, 129.09−130.67, 202.67. − Reduction of **27c** with TPP gave cyclopentanedicarbaldehyde [δ = 1.88−2.26 (m, 6 H), 2.83 (m, 2 H)] and benzoyl cyanide (**6c**) in a ratio of ca. 1:1.

Ozonolysis of Isopropenyl Acetate in the Presence of *trans*-27c: Ozonolysis of 0.82 g (3.0 mmol) of *trans*-**27c** and 0.60 g (6.0 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.23 g (0.72 mmol, 24.0%) of *trans*-**28c** was isolated [solvent: dichloromethane/*n*-pentane, 4:1].

5-Cyano-5-phenyl-3-[3-(1,2,4-trioxolan-3-yl)cyclopentyl]-1,2,4-trioxolane (*trans*-28c): Colorless liquid. − ^{1}H NMR: δ = 1.41−2.15 (m, 6 H), 2.32 (m, 1 H), 2.47 (m, 1 H), 4.77−5.00 (m, 2 H), 4.98 (s, 1 H), 5.00 (d, J = 5.24 Hz, 1 H), 5.11 (s, 1 H), 5.44 (d, J = 5.24 Hz, 1 H), 7.38−7.64 (m, 5 H). − ^{13}C NMR: δ = 26.61, 28.50, 29.16, 39.47, 40.50, 94.29, 101.11, 105.39, 109.79, 115.89, 127.02, 128.93, 132.03. − $C_{16}H_{17}NO_6$ (319.3): calcd. C 60.19, H 5.37; found C 59.84, H 5.21. − Reduction of **28c** with TPP gave cyclopentanedicarbaldehyde and benzoyl cyanide (**6c**) in a ratio of ca. 1:1.

Ozonolysis of 12 in the Presence of 6d: Ozonolysis of 0.74 g (8.0 mmol) of **12** and 1.05 g (24 mmol) of **6d** in 50 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.70 g (3.8 mmol, 47.8%) of *cis*- and *trans*-**27d** was isolated [solvent: dichloromethane/diethyl ether, 7:1].

(5-Methyl-1,2,4-trioxolan-3-yl)-3-cyclopentanecarbaldehyde (27d): Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.34 (d, *J* = 6.23 Hz, 3 H), 1.77−1.92 (m, 6 H), 2.23 (m, 1 H), 2.72 (m, 1 H), 5.08−5.11 (m, 1 H), [5.27 (q, *J* = 5.34 Hz), 5.33 (q, *J* = 5.34 Hz)] (1 H), 9.75 (s, 1 H). − ^{13}C NMR: δ = 16.49, 17.84, 26.32, 26.44, 27.19, 27.41, 27.52, 40.65, 40.88, 42.66, 51.48, 51.59, 101.60, 101.99, 106.26, 106.45, 203.14, 203.20. − $C_9H_{14}O_4$ (186.2): calcd. C 58.06, H 7.58; found C 58.45, H 7.71. − Reduction of **27d** with TPP gave cyclopentanedicarbaldehyde and acetaldehyde (**6d**) in a ratio of ca. 1:1.

Ozonolysis of Isopropenyl Acetate in the Presence of *cis*- and *trans*-27d: Ozonolysis of 0.74 g (4.0 mmol) of **27d** and 0.8 g (8.0 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a liquid residue, from which 0.35 g (1.5 mmol, 37.3%) of *cis*- and *trans*-**28d** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

***cis*- and *trans*-5-Methyl-3-[3-(1,2,4-trioxolan-3yl)cyclopentyl]-1,2,4-trioxolane (28d):** Colorless liquid (a mixture of *cis* and *trans* isomers). − ^{1}H NMR: δ = 1.32 (d, *J* = 6 .23 Hz, 3 H), 1.49−1.70 (m, 6 H), 2.15−2.18 (m, 2 H), 4.94 (m, 1 H), 4.96 (s, 1 H), 5.03 (t, *J* = 5.35 Hz, 1 H), 5.09 (s, 1 H), [5.24 (q, *J* = 5.12 Hz), 5.27 (q, *J* = 5.12 Hz)] (1 H). − ^{13}C NMR: δ = 9.48, 10.98, 13.79, 19.94, 20.05, 22.18, 22.21, 33.58, 33.88, 33.94, 35.20, 87.50, 94.58, 94.91, 99.51, 99.66. − $C_{10}H_{16}O_6$ (232.2): calcd. C 51.73, H 6.95; found C 51.38, H 6.83. − Reduction of **28d** with TPP gave cyclopentanedicarbaldehyde and acetaldehyde (**6d**) in a ratio of ca. 1:1.

Ozonolysis of 13 in the Presence of 6a: Ozonolysis of 0.46 g (3.0 mmol) of **13** and 1 mL of **6a** (freshly prepared by pyrolysis of paraformaldehyde) in 50 mL of dichloromethane at −78 °C, followed by distillation of the solvent under reduced pressure, gave a solid residue. The residue was recrystallized from dichloromethane to give 0.30 g (1.5 mmol, 50.2%) of **30**. From the filtrate, 0.1 g (0.48 mmol, 16.3%) of a colorless solid **29** was isolated by flash chromatography (silica gel, solvent: dichloromethane/*n*-pentane = 1:1).

8-Formyl-1-naphthalenecarboxylic Acid (Lactol Form, 30): Colorless needles; m.p. 167−168 °C (ref.[9][10] 167−168 °C). − ^{1}H NMR: δ = 4.62 (d, *J* = 6.25 Hz, 1 H), 6.94 (d, *J* = 6.25 Hz, 1 H), 7.64−7.74 (m, 3 H), 7.99 (d, *J* = 4.57 Hz, 1 H), 8.18 (d, *J* = 4.57 Hz, 1 H), 8.47 (d, *J* = 4.57 Hz, 1 H). − ^{13}C NMR: δ = 96.29, 119.28, 125.46, 127.12, 127.34, 128.12, 128.92, 129.12, 130.24, 132.14, 134.85, 164.61. − IR (KBr/film): $\tilde{\nu}$ = 3300 cm^{-1}, 1710, 1140.

Acenaphthylene Monoozonide (29): Colorless crystals; m.p. 102−103 °C (ref.[11] 102−105 °C). − ^{1}H NMR: δ = 6.69 (s, 2 H), 7.47−7.86 (m, 6 H). −^{13}C NMR: δ = 101.03, 122.12, 125.24, 126.28, 128.87, 131.69, 132.76. − Reduction of **29** with TPP gave 1,8-naphthalenedialdehyde [δ = 10.48 (s, 2 H)].

Ozonolysis of 13 in the Presence of 6b: Ozonolysis of 0.46 g (3.0 mmol) of **13** and 0.42 g (6 mmol) of pyruvonitrile **6b** in 50 mL of dichloromethane at −78 °C, followed by distillation of the solvent under reduced pressure, gave a solid residue. The residue was recrystallized from dichloromethane to give 0.36 g (1.80 mmol, 60.2%) of **30**. From the filtrate, 0.15 g (0.75 mmol, 25.3%) of crystalline **29** was isolated by flash chromatography (silica gel, solvent: dichloromethane/pentane = 1:1).

Ozonolysis of 13 in the Presence of 6c: Ozonolysis of 0.46 g (3.0 mmol) of **13** and 0.79 g (6 mmol) of **6c** in 50 mL of dichloromethane at −78 °C, followed by distillation of the solvent under reduced pressure, gave a solid residue. The residue was recrystallized from dichloromethane to give 0.36 g (1.80 mmol, 60.2%) of **30**. From the filtrate, 0.15 g (0.75 mmol, 25.3%) of crystalline **29** was isolated by flash chromatography (silica gel, solvent: dichloromethane/pentane = 1:1).

Ozonolysis of 14 in the Presence of 6a: Ozonolysis of 0.53 g (3 mmol) of phenanthrene **14** and 1 mL of **6a** (freshly prepared by pyrolysis of paraformaldehyde) in 50 mL of dichloromethane at −78 °C, followed by distillation of the solvent under reduced pressure, gave a liquid residue, from which 0.38 g (1.50 mmol, 49.0%) of **31a** was isolated [solvent: dichloromethane/*n*-pentane, 15:1].

2′-(1,2,4-Trioxolan-3-yl)biphenyl-2-carbaldehyde (31a): Colorless liquid (a mixture of two stereoisomers). − ^{1}H NMR: δ = 5.07 (s, 1 H), 5.21 (d, *J* = 9.13 Hz, 1 H), 5.78 (d, *J* = 12.56 Hz, 1 H), 7.24−8.03 (m, 8 H), 9.76 (d, *J* = 24.13 Hz, 1 H). − ^{13}C NMR: δ = 95.46, 95.62, 101.28, 101.68, 127.69−142.90 (m), 191.61, 191.99. The data are identical with those reported in the literature.[12] − Reduction of **31a** with TPP gave 5,6-biphenyldicarbaldehyde [^{1}H NMR: δ = 7.28−8.11 (m, 8 H), 9.86 (s, 2 H). − ^{13}C NMR: δ = 124.43−141.63, 191.48].

Ozonolysis of 14 in the Presence of 6b: Ozonolysis of 0.53 g (3 mmol) of phenanthrene **14** and 0.63 g (9 mmol) of **6b** in 50 mL of dichloromethane at −78 °C, followed by distillation of the solvent under reduced pressure, gave a liquid residue, from which 0.64 g (2.2 mmol, 74.3%) of **31b** was isolated [solvent: dichloromethane/*n*-pentane, 15:1].

2′-(5-Cyano-5-methyl-1,2,4-trioxolan-3-yl)biphenyl-2-carbaldehyde (31b): Colorless liquid (a mixture of stereoisomers). − ^{1}H NMR: δ = 1.94 (d, *J* = 12.23 Hz, 3 H), [5.85 (d, *J* = 6.12 Hz), 6.33 (d, *J* = 6.12 Hz)] (1 H), 7.30−8.08 (m, 8 H), [9.70 (t, *J* = 7.51 Hz), 9.80 (s)] (1 H). − ^{13}C NMR: δ = 20.55, 20.67, 20.86, 20.91, 97.96, 98.11, 99.28, 99.38, 102.51, 102.60, 103.18, 103.67, 115.45, 116.60, 127.74−141.38, 190.75, 190.99, 191.12, 191.15. − $C_{17}H_{13}NO_4$ (295.3): calcd. C 69.15, H 4.44; found C 68.84, H 4.31. − Reduction of **31b** with TPP gave 5,6-biphenyldicarbaldehyde and acetyl cyanide (**6b**) in a ratio of ca. 1:1.

Ozonolysis of 14 in the Presence of 6c: Ozonolysis of 0.53 g (3 mmol) of **14** and 1.18 g (9 mmol) of **6c** in 50 mL of dichloromethane at −78 °C, followed by distillation of the solvent under reduced pressure, gave a liquid residue, from which 0.79 g (2.2 mmol, 72.1%) of **31c** was isolated [solvent: dichloromethane/*n*-pentane, 2:1].

2′-(5-Cyano-5-phenyl-1,2,4-trioxolan-3-yl)biphenyl-2-carbaldehyde (31c): Colorless liquid (a mixture of stereoisomers). − ^{1}H NMR: δ = 6.54 (d, *J* = 6.15 Hz, 1 H), 7.37−8.17 (m, 13 H), [9.77 (d, *J* = 12.23 Hz), 9.87 (s)] (1 H). − ^{13}C NMR: δ = 101.84, 101.92, 104.43, 104.56, 115.95, 116.07, 126.90−141.38, 190.77, 191.07. − $C_{22}H_{15}NO_4$ (295.3): C 73.93, H 4.23; found C 72.63, H 4.12. − Reduction of **31c** with TPP gave 5,6-biphenyldicarbaldehyde and benzoyl cyanide (**6c**) in a ratio of ca. 1:1.

Ozonolysis of Isopropenyl Acetate in the Presence of 31a: Ozonolysis of 0.77 g (3.0 mmol) of **31a** and 0.60 g (6 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78 °C gave a solid residue, from which 0.57 g (1.9 mmol, 63.2%) of **32a** was isolated [solvent: dichloromethane/*n*-pentane, 2:1].

2,2′-Bis(1,2,4-trioxolane-3-yl)biphenyl (32a): Colorless solid; m.p. 93 °C (ref.[11] 94 °C). − ^{1}H NMR: δ = [4.99 (s), 5.04 (s), 5.17 (s), 5.20 (s), 5.22 (s), 5.26 (s), 5.28 (s), 5.30 (s)] (4 H), [5.72 (s), 5.76 (s), 5.79 (s), 5.83 (s)] (2 H), 7.27−7.76 (m, 8 H). − ^{13}C NMR: δ =

95.37, 95.47, 95.67, 95.72, 101.18, 101.27, 101.41, 101.56, 126.29−140.41. The data are identical with those reported in the literature.[11] − Reduction of **32a** with TPP gave 5,6-biphenyldicarbaldehyde.

Ozonolysis of Isopropenyl Acetate in the Presence of 31b: Ozonolysis of 0.88 g (3.0 mmol) of **31b** and 0.60 g (6 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78°C gave a solid residue, from which 0.65 g (1.9 mmol, 63.4%) of **32b** was isolated [solvent: dichloromethane/*n*-pentane, 15:1].

2-(5-Cyano-5-methyl-1,2,4-trioxolan-3-yl)-2′-(1,2,4-trioxolan-3-yl)-biphenyl (32b): Colorless liquid (a mixture of stereoisomers). − ^{1}H NMR: δ = 1.81−1.97 (m, 3 H), 4.92−5.29 (m, 2 H), [5.65−5.84 (m), 6.25−6.31(m)] (3 H), 7.19−7.95 (m, 8 H). − ^{13}C NMR: δ = 21.08, 21.18, 21.28, 21.36, 21.49, 95.51, 95.62, 95.69, 99.01, 99.09, 99.80, 99.97, 100.96, 101.23, 101.36, 101.49, 101.58, 103.13, 103.24, 103.69, 103.98 104.12, 116.21, 116.19, 117.32, 117.46, 128.11−141.11. − $C_{18}H_{15}NO_6$ (341.3): calcd. C 63.34, H 4.43; found C 63.87, H 4.35. − Reduction of **32b** with TPP gave 5,6-biphenyldicarbaldehyde and acetyl cyanide (**6b**) in a ratio of ca. 1:1.

Ozonolysis of Isopropenyl Acetate in the Presence of 31c: Ozonolysis of 1.07 g (3.0 mmol) of **31c** and 0.60 g (6 mmol) of isopropenyl acetate in 40 mL of dichloromethane gave a solid residue, from which 0.81 g (2.0 mmol, 66.0%) of **32c** was isolated [solvent: dichloromethane/*n*-pentane, 15:1].

2-(5-Cyano-5-phenyl-1,2,4-trioxolan-3-yl)-2′-(1,2,4-trioxolan-3-yl)-biphenyl (32c): Colorless liquid (a mixture of stereoisomers). − ^{1}H NMR: δ = 5.07−5.32 (m, 2 H), 6.17−6.69 (m, 2 H), 7.49−8.24 (m, 13 H). − ^{13}C NMR: δ = 95.64, 95.71, 97.19, 101.39, 101.27, 101.52, 101.70, 102.31, 102.61, 102.63, 105.14, 105.49, 105.63, 115.81, 115.99, 116.82, 116.88, 127.50−141.27. − $C_{23}H_{17}NO_6$ (403.4): calcd. C 68.48, H 4.25; found C 68.71, H 4.12. − Reduction of **32c** with TPP gave 5,6-biphenyldicarbaldehyde and benzoyl cyanide (**6c**) in a ratio of ca. 1:1.

Ozonolysis of 15 in the Presence of 6a: Ozonolysis of 0.61 g (3 mmol) of **15** and 1 mL of **6a** (freshly prepared by pyrolysis of paraformaldehyde) in 50 mL of dichloromethane at −78°C for 1 h (ca. 3 mmol of ozone), followed by distillation of the solvent under reduced pressure, gave a liquid residue, from which 0.45 g (1.60 mmol, 52.4%) of **33a** and 0.13 g (0.51 mmol, 17.1%) of **35** were isolated [solvent: dichloromethane/*n*-pentane, 2:1].

5-(1,2,4-Trioxolan-3-yl)phenanthrene-4-carbaldehyde (33a): Colorless liquid (a mixture of stereoisomers). − ^{1}H NMR: δ = 5.50 (s, 2 H), 6.55 (s, 1 H), 7.48−8.25 (m, 8 H), [10.09 (s), 10.23 (s)] (1 H). − ^{13}C NMR: δ = 95.85, 96.65, 101.70, 127.05−135.57, 191.19, 191.72. − IR (KBr/film): ṽ = 2900−3100 cm^{-1}, 1750. The data are identical to those reported in the literature.[12] − Reduction of **33a** with TPP gave 4,5-phenanthrene-8,9-dicarbaldehyde [^{1}H NMR: δ = 7.00−8.17 (m, 8 H), 10.03 (s, 2 H). − ^{13}C NMR: δ = 127.88−137.73, 190.92].

Pyrene Monoozonide (35): Colorless crystals; m.p. 162−164°C (ref.[12] 163−165°C). − ^{1}H NMR: δ = 6.67 (s, 2 H), 7.57−8.05 (m, 8 H). − ^{13}C NMR: δ = 106.63, 125.36, 126.20, 126.51, 127.81, 128.20, 129.18, 132.25, 135.06. The data are identical with those reported in the literature.[12] − Reduction of **35** with TPP gave 4,5-phenanthrene-8,9-dicarbaldehyde.

Ozonolysis of 15 in the Presence of 6b: Ozonolysis of 0.61 g (3 mmol) of pyrene **15** and 0.63 g (9 mmol) of **6b** (freshly prepared by pyrolysis of paraformaldehyde) in 50 mL of dichloromethane at −78°C for 1 h (ca. 3 mmol of ozone), followed by distillation of the solvent under reduced pressure, gave a liquid residue, from which 0.54 g (1.7 mmol, 62.3%) of **33b** and 0.17 g (0.66 mmol, 22.1%) of **35** were isolated [solvent: dichloromethane/*n*-pentane, 2:1].

5-(5-Cyano-5-methyl-1,2,4-trioxolan-3-yl)phenanthrene-4-carbaldehyde (33b): Colorless liquid (a mixture of stereoisomers). − ^{1}H NMR: δ = 1.17−2.08 (m, 3 H), [6.56 (s), 6.65 (s)] (1 H), 7.28−8.31 (m, 8 H), [10.00 (s), 10.14 (s)] (1 H). − ^{13}C NMR: δ = 20.96, 21.09, 99.06, 100.17, 103.71, 104.11, 116.86, 126.67−134.18, 190.42, 191.02. − $C_{19}H_{13}NO_4$ (319.3): calcd. C 71.47, H 4.10; found C (71.76), H 4.03. − Reduction of **33b** with TPP gave 4,5-phenanthrene-8,9-dicarbaldehyde and acetyl cyanide (**6b**) in a ratio of ca. 1:1.

Ozonolysis of Isopropenyl Acetate in the Presence of 33a: Ozonolysis of 0.84 g (3.0 mmol) of **33a** and 0.60 g (6 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78°C gave a solid residue, from which 0.39 g (1.2 mmol, 41.3%) of **34a** was isolated [solvent: dichloromethane/*n*-pentane, 2:1].

4,5-Di-[(1,2,4-trioxolan-3-yl)propyl]phenanthrene (34a): Colorless liquid (a mixture of stereoisomers). − ^{1}H NMR: δ = 5.27−5.62 (m, 4 H), 6.59−6.83 (m, 2 H), 7.60−8.03 (m, 8 H). − ^{13}C NMR: δ = 95.77, 96.68, 101.92, 102.15, 127.28−134.54. − $C_{18}H_{14}O_6$ (326.3): calcd. C 66.26, H 4.32; found C 66.38, H 4.57. − Reduction of **34a** with TPP gave 4,5-phenanthrene-8,9-dicarbaldehyde.

Ozonolysis of Isopropenyl Acetate in the Presence of 33b: Ozonolysis of 0.96 g (3.0 mmol) of **33b** and 0.60 g (6 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78°C gave a solid residue, from which 0.51 g (1.4 mmol, 46.3%) of **34b** was isolated [solvent: dichloromethane/*n*-pentane, 15:1].

5-(5-Cyano-5-methyl-1,2,4-trioxolan-3-yl)-4-(1,2,4-trioxolan-3-yl)-phenanthrene (34b): Colorless liquid (a mixture of stereoisomers). − ^{1}H NMR: δ = 1.71−2.10 (m, 3 H), 5.29−5.55 (m, 2 H), 6.46−6.93 (m, 2 H), 7.50−8.21 (m, 8 H). − ^{13}C NMR: δ = 21.64, 21.98, 22.03, 95.99, 96.49, 100.76, 101.77, 101.81, 104.58, 104.98, 117.44, 127.60−134. − $C_{20}H_{15}NO_6$ (365.3): calcd. C 65.76, H 4.14; found C 65.68, H 4.25. − Reduction of **34b** with TPP gave 4,5-phenanthrene-8,9-dicarbaldehyde and acetyl cyanide (**6d**).

Ozonolysis of 16 in the Presence of 6a: Ozonolysis of 0.57 g (3.0 mmol) of **16** and 1 mL of **6a** (freshly prepared by pyrolysis of paraformaldehyde) in 50 mL of dichloromethane at −78°C gave a mixture, which was kept at room temperature for 1 h. Subsequent distillation of the solvent under reduced pressure gave a solid residue. The residue was recrystallized from dichloromethane to give 0.33 g (1.38 mmol, 46.4%) of **40**. − After evaporation of the solvent from the cold reaction mixture, the residue was recrystallized with cold dichloromethane to give 0.62 g (2.46 mmol, 82%) of **36a**.

5-Formyl-4-fluorenecarboxylic Acid (40): Colorless crystals; m.p. 139−141°C (ref.[13] 140−142°C). − ^{1}H NMR ([D$_6$]acetone): δ = 3.95 (s, 2 H), 7.11−7.76 (m, 6 H), 10.14 (s, 1 H). − ^{13}C NMR ([D$_6$]acetone): δ = 37.23, 170.00, 190.38.

5-(1,2,4-Trioxolan-3-yl)fluorene-4-carbaldehyde (36a): Colorless crystals; m.p. 100−102°C. − ^{1}H NMR: δ = 4.01 (s, 2 H), 5.30 (s, 1 H), 5.40 (s, 1 H), 6.35 (s, 1 H), 7.45−7.86 (m, 6 H), 10.44 (s, 1 H). − ^{13}C NMR: δ = 37.73, 95.62, 101.45, 126.75−145.90 (m), 193.04. − $C_{16}H_{12}O_4$ (268.3): calcd. C 71.63, H 4.51; found C 71.44, H 4.65. − Reduction of **36a** with TPP gave 4,5-methylenephenanthrene-8,9-dicarbaldehyde [^{1}H NMR: δ = 3.91 (s, 2 H), 10.07 (s, 2 H). − ^{13}C NMR: δ = 37.46, 190.99].

Ozonolysis of 16 in the Presence of 6b: Ozonolysis of 0.57 g (3.0 mmol) of **16** and 0.42 g (6 mmol) of pyruvonitrile **6b** in 50 mL of dichloromethane at −78°C, followed by distillation of the solvent under reduced pressure, gave a solid residue. The residue was recrystallized from dichloromethane to give 0.32 g (1.35 mmol, 45.3%) of **40**. From the filtrate, 0.09 g (0.39 mmol, 13.1%) of **39** was isolated by flash chromatography (silica gel, solvent: dichloromethane/pentane, 1:1). − Another reaction mixture of this ozonolysis was admixed with a solution of 0.5 g (6 mmol) of *O*-methylhydroxylamine and 1 mL of pyridine in 10 mL of methanol (precooled to −20°C), and the mixture was kept at low temperature for several days. The solvent was then evaporated at room temperature and under reduced pressure, the residue was admixed with water, and the mixture was extracted with ether. The extract was sequentially washed with 2 M aqueous hydrochloric acid and an aqueous solution of sodium bicarbonate, dried with $MgSO_4$, filtered, and concentrated by evaporation of the solvent at room temperature and under reduced pressure. From this residue 0.12 g (0.36 mmol, 12.1%) of **37b** was isolated (silica gel, solvent: *n*-pentane/ether, 20:1).

4,5-Methylenephenanthrene Monoozonide (39): Colorless crystals; m.p. 141−142°C (ref.[13] 141−143°C). − ^{1}H NMR: δ = 3.94 (s, 2 H), 6.46 (s, 2 H), 7.24−7.59 (m, 6 H). − ^{13}C NMR: δ = 37.56, 104.26, 125.85, 126.67, 127.40, 132.48, 139.96, 144.36. The data are identical with those reported in the literature. − Reduction of **39** with TPP gave 4,5-methylenephenanthrene-8,9-dicarbaldehyde.

5-(5-Cyano-5-methyl-1,2,4-trioxolan-3yl)fluorene-4-carbaldehyde *O*-Methyloxime (37b): Colorless crystals; m.p. 103−104°C. − ^{1}H NMR: δ = 2.00 (s, 3 H), 3.91 (s, 2 H), 4.02 (s, 3 H), 6.66 (s, 1 H), 7.24−7.95 (m, 6 H), 8.65 (s, 1 H). − ^{13}C NMR: δ = 21.68, 37.76, 62.34, 100.25, 104.02, 117.13, 150.01. − $C_{19}H_{16}N_2O_4$ (336.4): calcd. C 67.84, H 4.79; found C 68.07, H 4.74.

Ozonolysis of 16 in the Presence of 6c: Ozonolysis of 0.57 g (3.0 mmol) of **16** and 0.79 g (6 mmol) of benzoyl cyanide **6c** in 50 mL of dichloromethane at −78°C, followed by distillation of the solvent under reduced pressure, gave a liquid residue, from which 0.46 g (1.92 mmol, 64.3%) of **39** was isolated by flash chromatography (silica gel, solvent: dichloromethane/*n*-pentane, 1:1).

Ozonolysis of Isopropenyl Acetate in the Presence of 36a: Ozonolysis of 0.76 g (3.0 mmol) of **36a** and 0.60 g (6 mmol) of isopropenyl acetate in 40 mL of dichloromethane at −78°C gave a solid residue, from which 0.25 g (0.78 mmol, 26.2%) of **38a** was isolated [solvent: dichloromethane/diethyl ether, 15:1].

4,5-Bis(2,4,6-trioxolan-3-yl)fluorene (38a): Colorless solid; m.p. 95−95°C. − ^{1}H NMR: δ = 3.90 (s, 2 H), 5.42 (s, 1 H), 5.44 (s, 1 H), 5.50 (s,1 H), 5.51 (s, 1 H), 6.74 (s, 1 H), 6.75 (s, 1 H), 7.30−7.80 (m, 6 H). − ^{13}C NMR: δ = 37.85, 96.03, 101.27, 127.14, 127.28, 127.83, 127.94, 140.62, 145.38. − $C_{17}H_{14}O_6$ (314.3): calcd. C 64.97, H 4.49; found C 64.71, H 4.38. − Reduction of **38a** with TPP gave 4,5-methylenephenanthrene-8,9-dicarbaldehyde.

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