SUPPORTING INFORMATION:

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"Functionalized Derivatives of 1,4-Dimethylnaphthalene as Precursors for Biomedical Applications : Synthesis, Structures, Spectroscopy and Photochemical Activation"

Formation of Ethylcellulose (EC) Nanoparticles

Ethylcellulose was commercially available from Sigma-Aldrich, (Munich, Germany). EC nanoparticles were prepared via a precipitation procedure from a mixture of solvents. To 20 mL of 0.2 % (w/w) of ethylcellulose in acetone/tetrahydrofuran = 1/1, 40 mL of ddH_2O (non-solvent) were poured fast to form ethylcellulose nanoparticles. Acetone and THF were subsequently evaporated from such a suspension of ethylcellulose particles in ddH_2O /acetone/THF on a rotary evaporator, resulting in EC nanoparticles suspended in ddH_2O (cca. 0.1 % w/w) with a diameter of 62 ± 18 nm (measured with dynamic light scattering). The EC nanoparticle concentration in aqueous suspension (w/w) was determined gravimetrically: by measuring the mass of the EC aqueous suspension and the mass of the EC left after careful water evaporation using a rotary evaporator at 55 °C.

Doping of EC Nanoparticles with DMN-derivatives and Dyes

In order to load EC particles with DMNOH (**2**), 10.0 mg of (**2**) in acetone were added to 20 mL of 0.2 % (w/w) solution of EC in acetone/tetrahydrofuran = 1/1 and stirred for 10 minutes at room temperature. Then 40 mL of dd H_2O were poured fast into this „cocktail" to get ethylcellulose nanoparticles. Acetone and THF were subsequently evaporated with a rotary evaporator, resulting in EC nanoparticles doped with (**2**) **(**diameter of 98 ± 25 nm, measured with DLS) and (**2**) concentration of $8.2 \cdot 10^{-4}$ M in aqueous suspension of EC nanoparticle carrier. A very similar procedure was done to prepare an aqueous suspension of (**3**) embedded in EC nanoparticles. A slightly different outcome was in the average diameter of the particles (136 ± 38 nm) and concentration in dd H_2O suspension ($4.2 \cdot 10^{-4}$ M) for (**3**).

The concentration of doped naphthalene derivatives (**2**,**3**) aqueous suspension of EC nanoparticles was determined spectroscopically with a standard addition method according to

Lambert-Beer's law. For each naphthalene-derivative a set of absorbance measurements in UV region at the corresponding peak maximum wavelength, respectively, was done. Prior to the measurement EC nanoparticles were dissolved in methanol in order to prevent light scattering on nanoparticles. From each sample three various aliquots were diluted in methanol and measured to check the fulfilling of Lambert-Beer's law linear dependence of absorbance on sample concentration. A calibration curve was made with a standard solution of known concentration of a particular naphthalene-derivative in methanol.

For confocal laser microscopy experiments (see below) EC nanoparticles doped with *N*,*N'*-bis(2,6 -dimethylphenyl)- perylene-3,4,9,10- tetracarboxylic diimide (PTC) were prepared:
EC and PTC (1% w/w to EC weight) were dissolved in 20 mL acetone/THF=1/1 and 40 mL of dd H_2O were poured fast into this „cocktail" to get EC nanoparticles. Acetone and THF were subsequently evaporated with a rotary evaporator, resulting in EC nanoparticles doped with PTC.

Endoperoxide Generation within EC Nanoparticles

Methylene blue (MB) as a photosensitizer dissolved in H_2O was added to the prepared aqueous suspension of naphthalene-derivatives embedded in EC nanoparticles (a 10^{-5} M concentration of MB in dd H_2O was used). Afterwards this suspension was irradiated with red laser light (658 nm; 70 mW; 140 $mWcm^{-2}$) for various periods of time at different temperatures (e.g. 15 h at 4 °C) to form the corresponding endoperoxides (**4**) and (**5**) inside the EC nanoparticles.
These samples of endoperoxides embedded in EC nanoparticles (aqueous suspensions) were tested for endoperoxide thermolysis and $^1O_2$-release or kinetic chemosensitivity assay on human breast cancer cells. Furthermore, after the irradiation and just prior to the chemosensitivity assay, polysorbate 80™ surfactant (by Sigma-Aldrich, Munich, Germany) was added to the EC nanoparticle suspensions (1% w/w end concentration of polysorbate 80™ in dd H_2O) in order to enable faster uptake of the EC nanoparticles by the breast cancer cells. The samples were left for 5 minutes in an ultrasonic bath to promote the adsorption of polysorbate 80™ on the EC nanoparticle surface.

Confocal Laser Scanning Microscopy (CLSM)

Two days prior to the experiment, the MDA-MB-231 cells (100 % confluency) were trypsinized and seeded in Nunc LabTek™ II chambered cover glasses with 8 chambers in RPMI 1640 or McCoy´s 5A medium (250 µl) that contained 5 % FCS. On the day of the experiment the confluency of the cells was 40-50 %. After the culture medium was removed, the cells were washed once with Leibowitz's L-15 culture medium (Invitrogen GmbH, Darmstadt, Germany). Subsequently, the cells were stained in L-15 medium containing 2.5 µg/ml CellMask™ Deep Red plasma membrane stain for 5 minutes at 37 °C. This staining solution was removed and the cells were washed 3 times with L-15 medium. Finally, the cells were covered with 250 µl of L-15 medium containing a 5 µM DRAQ5™ nuclear stain and suspension of polymer nanoparticles doped with *N*,*N'*-bis(2,6 -dimethylphenyl)- perylene-3,4,9,10- tetracarboxylic diimide (PTC). The polymer nanoparticle suspension was diluted 1:50 with L-15 medium. This photosensitizer was chosen to label the polymer nanoparticles because of a high molar absorption coefficient at available CLSM Ar^+-laser wavelengths (488 nm, 514 nm): ε(488 nm) = 4.55 •10^4 $cm^{-1}M^{-1}$, ε(514 nm) = 3.05 •10^4 $cm^{-1}M^{-1}$, which is critical for fluorescence microscopy recording of small polymer nanoparticles (diameter < 200nm).

Confocal laser scanning microscopy experiments were performed with a Carl Zeiss Axiovert 200M microscope (Carl Zeiss AG, Oberkochen, Germany), equipped with a LSM 510 laser scanner. PVB and EC nanoparticles (stained with PTC) were detected through the 530-600 nm band-pass filter, after excitation with the 488 nm laser (4.8 % laser transmission). The nuclei stained with DRAQ5™ and the plasma membranes stained with CellMask™ Deep Red were detected with the 650 nm long-pass filter after excitation with the 633 nm laser (3.1 % laser transmission). The objective used was a Plan-Apochromat 63R/1.4 with Immersol™ 518 F immersion oil for fluorescence microscopy (Carl Zeiss AG, Oberkochen, Germany).

Cell Line and Culture Conditions

Human estrogen receptor negative MDA-MB-231 (HTB 26) breast cancer cells were obtained from the American Type Culture Collection (ATCC), Rockville, USA. Cell banking and quality control were performed according to the "seed stock concept". Cells were cultured in RPMI 1640 or McCoy´s 5A medium (Sigma Aldrich, Munich, Germany) containing L-glutamine, 2.2 g/l $NaHCO_3$ and 5 % fetal calf serum, FCS (Biochrom, Berlin,

Germany). Cells were maintained in a water saturated atmosphere (95 % air / 5 % carbon dioxide) at 37°C in 75-cm² culture flasks (Greiner, Frickenhausen, Germany), and were serially passaged following trypsinization using 0.05% trypsin/0.02% EDTA (Roche Diagnostics, Mannheim, Germany). *Mycoplasma* contamination was routinely monitored, and only Mycoplasma free cultures were used.

Chemosensitivity Assays

The crystal violet chemosensitivity assays were performed with MDA-MB-231 human breast cancer cells according to a published procedure [Bernhardt G, Reile H, Birnböck H, Spruss T, Schönenberger H, "Standardized kinetic microassay to quantify differential chemosensitivity on the basis of proliferative activity", *J Cancer Res Clin Oncol* 1992, *118*, 35–43].

Tumor cell suspensions (100 µl/well) were seeded into 96-well flat bottomed microtitration plates (Greiner, Frickenhausen, Germany) at a density of 10-15 cells/microscopic field (magnification 32x). After 1-2 days the McCoy´s 5A or RPMI 1640 culture medium (containing 5 % FCS) was removed by suction and replaced with fresh medium (200 µl/well) containing varying drug concentrations or different polymer carrier formulations, respectively.

On every plate 16 wells served as controls and 16 wells were used per drug concentration. After various times of incubation the cells were fixed with glutardialdehyde and stored in a refrigerator. At the end of the experiment all plates were stained with crystal violet simultaneously. Absorbance, which corresponds to the living cells mass at each time-point, was measured at 578 nm using a Biotek 309 Autoreader (Tecnomara, Fernwald, Germany). The absorbance values were transformed into corrected T/C values, expressing the net growth of the treated cells, relative to the growth of the untreated control cells. Corrected T/C values were calculated according to equation

$$(T/C)_{corr}[\%] = \frac{A_T - A_{c,0}}{A_c - A_{c,0}} \cdot 100$$

where A_T is the mean absorbance of the treated cells, A_C the mean absorbance of the controls and Ac,o the mean absorbance at the time (t = 0) when drug was added.

In this context drug fomulations used were endoperoxides of various naphthalene derivatives embedded in various carrier materials (e.g. EC-nanoparticles coated with Polysorbate-80™ or

non-coated), which were suspended in double distilled water. In order to differentiate the effect of endoperoxides from the possible effect of their "parent molecules" (naphthalene derivatives) and/or carrier materials on the proliferation of MDA-MB-231 cells, for each aromatic endoperoxides drug formulation, two sets of control formulations were used:

1. non-irradiated undoped carrier material suspension of the same concentration (in ddH_2O) as in the drug formulation to determine the carrier effect on the cell proliferation and

2. non-irradiated naphthalene "parent molecule" of the same concentration as the endoperoxide and the same photosensitizer concentration (as in the drug formulation) embedded in the same carrier material suspension, also of the same concentration as in the drug formulation to determine the photosensitizer and "parent molecule" effect on the cell proliferation.

Prior to the cell incubation, each of these formulations was sterilised for 15 min by irradiation with a UV-A lamp. As a positive control of the proliferation drug effect on the MDA-MB-231 cells, the clinically established cytostatics cisplatin (Sigma-Aldrich, Deisenhofen, Germany) or vinblastine (Sigma-Aldrich, Deisenhofen, Germany) were used.

Crystallographic Data for Compounds (2) and (3):

Table 1. Crystal data and structure refinement for 773209, DMNOH (2):

Crystal Data ;

Empirical formula ;C14 H16 O
Formula weight ;200.27

Crystal size ;0.210 x 0.042 x 0.008 mm

Crystal description ;elongated plate

Crystal colour ;colourless

Crystal system;Monoclinic

Space group ;P 21/n

Unit cell dimensions ;a = 8.1442(3) A alpha = 90 deg.
;b = 4.8533(2) A beta = 94.010(4) deg.
;c = 27.8100(11) A gamma = 90 deg.

Volume ;1096.53(7) A^3

Z, Calculated density ;4, 1.213 Mg/m^3

Absorption coefficient ;0.574 mm^-1

F(000) ;432

Data Collection ;

Measurement device type ;Goniometer Xcalibur, detector: Ruby (Gemini ultra Mo)

Measuremnet method ;\w scans

Temperature ;123 K

Wavelength ;1.54184 A

Monochromator ; graphite

Theta range for data collection ;3.19 to 66.33 deg.

Index ranges ;-9<=h<=7, -5<=k<=5, -29<=l<=32

Reflections collected / unique ;4568 / 1897 [R(int) = 0.0247]

Reflections greater I>2\s(I);1574

Absorption correction ;Semi-empirical from equivalents

Max. and min. transmission ;1.00000 and 0.84432

Refinement ;

Refinement method ;Full-matrix least-squares on F^2

Hydrogen treatment ;mixed

Data / restraints / parameters ;1897 / 0 / 143

Goodness-of-fit on F^2 ;1.051

Final R indices [I>2sigma(I)] ;R1 = 0.0392, wR2 = 0.1094

R indices (all data) ;R1 = 0.0464, wR2 = 0.1136

Absolute structure parameter ;.

Largest diff. peak and hole ;0.171 and -0.223 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for i191.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

;x ;y ;z ;U(eq)

O(1);2901(1);-3253(2);7306(1);31(1)
C(1);2704(2);-3203(3);6790(1);28(1)
C(2);1116(2);-1752(3);6621(1);37(1)
C(3);4177(2);-1906(2);6567(1);24(1)
C(4);5110(2);94(2);6839(1);25(1)
C(5);6423(2);1457(3);6668(1);25(1)
C(6);6863(2);848(3);6190(1);27(1)
C(7);8162(2);2244(3);5979(1);33(1)
C(8);8552(2);1687(3);5517(1);41(1)
C(9);7664(2);-314(3);5245(1);40(1)
C(10);6401(2);-1698(3);5435(1);34(1)
C(11);5938(2);-1168(3);5911(1);27(1)
C(12);4581(2);-2558(3);6107(1);26(1)
C(13);7372(2);3526(3);6978(1);31(1)
C(14);3627(2);-4677(3);5801(1);33(1)

Table 3. Bond lengths [A] and angles [deg] for i191.

O(1)-C(1);1.4330(16)
O(1)-H(1O);0.89(2)
C(1)-C(3);1.5247(18)
C(1)-C(2);1.5180(19)
C(3)-C(12);1.3807(16)
C(3)-C(4);1.4180(15)
C(4)-C(5);1.3716(17)
C(5)-C(13);1.5021(19)
C(5)-C(6);1.4312(16)
C(6)-C(7);1.4181(19)
C(6)-C(11);1.4302(19)
C(7)-C(8);1.370(2)
C(8)-C(9);1.401(2)
C(9)-C(10);1.366(2)
C(10)-C(11);1.4231(18)
C(11)-C(12);1.4350(18)
C(12)-C(14);1.5144(19)
C(1)-H(1);1.0000
C(2)-H(2A);0.9800
C(2)-H(2B);0.9800
C(2)-H(2C);0.9800
C(4)-H(4);0.9500
C(7)-H(7);0.9500
C(8)-H(8);0.9500
C(9)-H(9);0.9500
C(10)-H(10);0.9500
C(13)-H(13A);0.9800
C(13)-H(13B);0.9800
C(13)-H(13C);0.9800
C(14)-H(14A);0.9800
C(14)-H(14B);0.9800
C(14)-H(14C);0.9800

C(1)-O(1)-H(1O);109.2(13)
O(1)-C(1)-C(2);110.60(11)
C(2)-C(1)-C(3);111.11(11)
O(1)-C(1)-C(3);112.18(10)
C(1)-C(3)-C(4);118.23(10)
C(1)-C(3)-C(12);121.83(11)
C(4)-C(3)-C(12);119.90(11)
C(3)-C(4)-C(5);123.10(11)
C(4)-C(5)-C(6);118.37(11)
C(6)-C(5)-C(13);121.28(11)
C(4)-C(5)-C(13);120.36(10)
C(5)-C(6)-C(11);119.28(11)
C(7)-C(6)-C(11);118.80(11)
C(5)-C(6)-C(7);121.89(12)
C(6)-C(7)-C(8);121.49(13)

C(7)-C(8)-C(9);119.87(13)
C(8)-C(9)-C(10);120.37(13)
C(9)-C(10)-C(11);121.79(13)
C(6)-C(11)-C(12);120.38(10)
C(10)-C(11)-C(12);121.95(12)
C(6)-C(11)-C(10);117.67(12)
C(3)-C(12)-C(14);122.08(11)
C(11)-C(12)-C(14);118.95(10)
C(3)-C(12)-C(11);118.97(11)
O(1)-C(1)-H(1);108.00
C(2)-C(1)-H(1);108.00
C(3)-C(1)-H(1);108.00
C(1)-C(2)-H(2A);109.00
C(1)-C(2)-H(2B);109.00
C(1)-C(2)-H(2C);109.00
H(2A)-C(2)-H(2B);110.00
H(2A)-C(2)-H(2C);109.00
H(2B)-C(2)-H(2C);109.00
C(3)-C(4)-H(4);118.00
C(5)-C(4)-H(4);118.00
C(6)-C(7)-H(7);119.00
C(8)-C(7)-H(7);119.00
C(7)-C(8)-H(8);120.00
C(9)-C(8)-H(8);120.00
C(8)-C(9)-H(9);120.00
C(10)-C(9)-H(9);120.00
C(9)-C(10)-H(10);119.00
C(11)-C(10)-H(10);119.00
C(5)-C(13)-H(13A);109.00
C(5)-C(13)-H(13B);109.00
C(5)-C(13)-H(13C);109.00
H(13A)-C(13)-H(13B);109.00
H(13A)-C(13)-H(13C);109.00
H(13B)-C(13)-H(13C);109.00
C(12)-C(14)-H(14A);109.00
C(12)-C(14)-H(14B);110.00
C(12)-C(14)-H(14C);110.00
H(14A)-C(14)-H(14B);109.00
H(14A)-C(14)-H(14C);110.00
H(14B)-C(14)-H(14C);109.00

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for i191.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

;U11 ;U22 ;U33 ;U23 ;U13 ;U12

O(1);41(1);24(1);30(1);2(1);12(1);1(1)
C(1);34(1);21(1);30(1);-1(1);5(1);-3(1)
C(2);28(1);36(1);47(1);2(1);4(1);-5(1)
C(3);27(1);19(1);27(1);2(1);2(1);4(1)
C(4);29(1);21(1);25(1);1(1);4(1);5(1)
C(5);25(1);21(1);29(1);2(1);2(1);4(1)
C(6);26(1);24(1);31(1);5(1);4(1);7(1)
C(7);27(1);33(1);39(1);7(1);6(1);5(1)
C(8);34(1);45(1);44(1);14(1);16(1);9(1)
C(9);46(1);45(1);31(1);5(1);15(1);15(1)
C(10);40(1);34(1);28(1);1(1);6(1);11(1)
C(11);33(1);24(1);26(1);3(1);5(1);9(1)
C(12);32(1);20(1);27(1);2(1);1(1);5(1)
C(13);30(1);27(1);37(1);-1(1);2(1);-1(1)
C(14);43(1);27(1);30(1);-4(1);0(1);2(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for i191.

;x ;y ;z ;U(eq)

H(1);2617;-5155;6676;34
H(1O);2640(20);-1610(50);7420(7);53(5)
H(2A);190;-2624;6769;55
H(2B);950;-1888;6269;55
H(2C);1183;192;6715;55
H(4);4810;510;7155;30
H(7);8776;3595;6161;39
H(8);9423;2657;5382;49
H(9);7942;-710;4926;48
H(10);5816;-3051;5245;41
H(13A);6848;3755;7282;47
H(13B);7384;5298;6809;47
H(13C);8504;2876;7045;47
H(14A);2832;-5601;5995;50
H(14B);4391;-6042;5683;50
H(14C);3041;-3761;5525;50

Table 6. Torsion angles [deg] for i191.

O(1)-C(1)-C(3)-C(4);-28.09(15)
O(1)-C(1)-C(3)-C(12);154.20(11)
C(2)-C(1)-C(3)-C(4);96.30(13)
C(2)-C(1)-C(3)-C(12);-81.42(15)
C(1)-C(3)-C(4)-C(5);-177.74(11)
C(12)-C(3)-C(4)-C(5);0.02(18)
C(1)-C(3)-C(12)-C(11);177.16(12)
C(1)-C(3)-C(12)-C(14);-2.33(19)
C(4)-C(3)-C(12)-C(11);-0.51(18)
C(4)-C(3)-C(12)-C(14);-180.00(12)
C(3)-C(4)-C(5)-C(6);0.60(18)
C(3)-C(4)-C(5)-C(13);-179.17(11)
C(4)-C(5)-C(6)-C(7);177.44(12)
C(4)-C(5)-C(6)-C(11);-0.71(19)
C(13)-C(5)-C(6)-C(7);-2.8(2)
C(13)-C(5)-C(6)-C(11);179.06(12)
C(5)-C(6)-C(7)-C(8);-178.64(13)
C(11)-C(6)-C(7)-C(8);-0.5(2)
C(5)-C(6)-C(11)-C(10);179.38(12)
C(5)-C(6)-C(11)-C(12);0.2(2)
C(7)-C(6)-C(11)-C(10);1.17(19)
C(7)-C(6)-C(11)-C(12);-177.98(13)
C(6)-C(7)-C(8)-C(9);-0.4(2)
C(7)-C(8)-C(9)-C(10);0.6(2)
C(8)-C(9)-C(10)-C(11);0.2(2)
C(9)-C(10)-C(11)-C(6);-1.1(2)
C(9)-C(10)-C(11)-C(12);178.09(13)
C(6)-C(11)-C(12)-C(3);0.38(19)
C(6)-C(11)-C(12)-C(14);179.89(12)
C(10)-C(11)-C(12)-C(3);-178.73(12)
C(10)-C(11)-C(12)-C(14);0.8(2)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen-bonds for i191 [A and deg.].

D-H...A;d(D-H);d(H...A);d(D...A);<(DHA)

O(1)-H(1O)...O(1)#1; 0.89(2); 1.87(2); 2.7531(13); 177(2)
C(4)-H(4)...O(1); 0.9500; 2.4500; 2.8080(15); 102.00

Table 2. Crystal data and structure refinement for 773208, DMNOH (3):

Crystal Data ;

Empirical formula ;C28 H30 O
Formula weight ;382.52

Crystal size ;0.270 x 0.190 x 0.050 mm

Crystal description ;plate

Crystal colour ;translucent, colourless

Crystal system;Orthorhombic

Space group ;P b c n

Unit cell dimensions ;a = 7.6851(3) A alpha = 90 deg.
;b = 16.8705(5) A beta = 90 deg.
;c = 16.3611(5) A gamma = 90 deg.

Volume ;2121.24(12) A^3

Z, Calculated density ;4, 1.198 Mg/m^3

Absorption coefficient ;0.536 mm^-1

F(000) ;824

Data Collection ;

Measurement device type ;Goniometer Xcalibur, detector: Ruby (Gemini ultra Mo)

Measuremnet method ;\w scans

Temperature ;123 K

Wavelength ;1.54184 A

Monochromator ; graphite

Theta range for data collection ;5.24 to 65.05 deg.

Index ranges ;-8<=h<=6, -17<=k<=19, -9<=l<=18

Reflections collected / unique ;6017 / 1772 [R(int) = 0.0210]

Reflections greater I>2\s(I);1511

Absorption correction ;Semi-empirical from equivalents

Max. and min. transmission ;1.00000 and 0.88389

Refinement ;

Refinement method ;Full-matrix least-squares on F^2

Hydrogen treatment ;:

Data / restraints / parameters ;1772 / 0 / 133

Goodness-of-fit on F^2 ;1.059

Final R indices [I>2sigma(I)] ;R1 = 0.0444, wR2 = 0.1234

R indices (all data) ;R1 = 0.0510, wR2 = 0.1282

Absolute structure parameter ;.

Largest diff. peak and hole ;0.263 and -0.225 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for i146.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	;x	;y	;z	;U(eq)
O(1)	5000	636(1)	7500	30(1)
C(1)	2752(2)	1616(1)	7188(1)	28(1)
C(2)	2497(2)	2422(1)	7270(1)	29(1)
C(3)	1776(2)	2854(1)	6594(1)	29(1)
C(4)	1561(2)	3687(1)	6616(1)	34(1)
C(5)	910(2)	4099(1)	5963(1)	37(1)
C(6)	401(2)	3698(1)	5253(1)	37(1)
C(7)	558(2)	2892(1)	5210(1)	33(1)
C(8)	1261(2)	2450(1)	5867(1)	28(1)
C(9)	1458(2)	1607(1)	5820(1)	29(1)
C(10)	2206(2)	1227(1)	6468(1)	29(1)
C(11)	3603(2)	1101(1)	7839(1)	30(1)
C(12)	2331(2)	515(1)	8212(1)	38(1)
C(13)	2932(2)	2859(1)	8051(1)	37(1)
C(14)	859(2)	1155(1)	5080(1)	36(1)

Table 3. Bond lengths [A] and angles [deg] for i146.

O(1)-C(11);1.4416(18)
O(1)-C(11)#1;1.4416(18)
C(1)-C(2);1.382(2)
C(1)-C(10);1.412(2)
C(1)-C(11);1.523(2)
C(2)-C(3);1.435(2)
C(2)-C(13);1.512(2)
C(3)-C(4);1.416(2)
C(3)-C(8);1.427(2)
C(4)-C(5);1.370(2)
C(5)-C(6);1.399(2)
C(6)-C(7);1.366(2)
C(7)-C(8);1.416(2)
C(8)-C(9);1.432(2)
C(9)-C(10);1.366(2)
C(9)-C(14);1.504(2)
C(11)-C(12);1.518(2)
C(4)-H(4);0.9500
C(5)-H(5);0.9500
C(6)-H(6);0.9500
C(7)-H(7);0.9500
C(10)-H(10);0.9500
C(11)-H(11);1.0000
C(12)-H(12A);0.9800
C(12)-H(12B);0.9800
C(12)-H(12C);0.9800
C(13)-H(13A);0.9800
C(13)-H(13B);0.9800
C(13)-H(13C);0.9800
C(14)-H(14A);0.9800
C(14)-H(14B);0.9800
C(14)-H(14C);0.9800

C(11)-O(1)-C(11)#1;114.02(14)
C(2)-C(1)-C(10);119.80(14)
C(2)-C(1)-C(11);123.64(14)
C(10)-C(1)-C(11);116.56(14)
C(1)-C(2)-C(3);118.65(14)
C(1)-C(2)-C(13);122.04(14)
C(3)-C(2)-C(13);119.31(14)
C(2)-C(3)-C(4);121.95(15)
C(2)-C(3)-C(8);120.49(14)
C(4)-C(3)-C(8);117.56(14)
C(3)-C(4)-C(5);121.80(15)
C(4)-C(5)-C(6);120.23(16)
C(5)-C(6)-C(7);120.01(16)
C(6)-C(7)-C(8);121.21(15)
C(3)-C(8)-C(7);119.17(15)

C(3)-C(8)-C(9);119.29(14)
C(7)-C(8)-C(9);121.55(15)
C(8)-C(9)-C(10);118.03(14)
C(8)-C(9)-C(14);120.93(14)
C(10)-C(9)-C(14);121.05(15)
C(1)-C(10)-C(9);123.62(15)
O(1)-C(11)-C(1);111.12(12)
O(1)-C(11)-C(12);106.22(13)
C(1)-C(11)-C(12);112.07(13)
C(3)-C(4)-H(4);119.00
C(5)-C(4)-H(4);119.00
C(4)-C(5)-H(5);120.00
C(6)-C(5)-H(5);120.00
C(5)-C(6)-H(6);120.00
C(7)-C(6)-H(6);120.00
C(6)-C(7)-H(7);119.00
C(8)-C(7)-H(7);119.00
C(1)-C(10)-H(10);118.00
C(9)-C(10)-H(10);118.00
O(1)-C(11)-H(11);109.00
C(1)-C(11)-H(11);109.00
C(12)-C(11)-H(11);109.00
C(11)-C(12)-H(12A);109.00
C(11)-C(12)-H(12B);109.00
C(11)-C(12)-H(12C);109.00
H(12A)-C(12)-H(12B);109.00
H(12A)-C(12)-H(12C);110.00
H(12B)-C(12)-H(12C);109.00
C(2)-C(13)-H(13A);109.00
C(2)-C(13)-H(13B);109.00
C(2)-C(13)-H(13C);109.00
H(13A)-C(13)-H(13B);109.00
H(13A)-C(13)-H(13C);109.00
H(13B)-C(13)-H(13C);109.00
C(9)-C(14)-H(14A);110.00
C(9)-C(14)-H(14B);109.00
C(9)-C(14)-H(14C);109.00
H(14A)-C(14)-H(14B);110.00
H(14A)-C(14)-H(14C);109.00
H(14B)-C(14)-H(14C);109.00

Symmetry transformations used to generate equivalent atoms:
#1 1/2-x+1,1/2-y,1/2+z+1

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for i146.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

;U11 ;U22 ;U33 ;U23 ;U13 ;U12

O(1);20(1);30(1);40(1);0;3(1);0
C(1);20(1);33(1);32(1);-2(1);3(1);0(1)
C(2);19(1);34(1);33(1);-5(1);0(1);1(1)
C(3);19(1);33(1);35(1);-4(1);2(1);0(1)
C(4);28(1);33(1);41(1);-7(1);-2(1);1(1)
C(5);29(1);31(1);51(1);0(1);-1(1);2(1)
C(6);28(1);40(1);42(1);7(1);-2(1);3(1)
C(7);25(1);41(1);33(1);-1(1);-1(1);-2(1)
C(8);18(1);34(1);33(1);-2(1);3(1);-1(1)
C(9);22(1);33(1);32(1);-3(1);3(1);-4(1)
C(10);24(1);28(1);36(1);-3(1);4(1);-1(1)
C(11);23(1);34(1);33(1);-2(1);2(1);4(1)
C(12);27(1);45(1);41(1);8(1);5(1);3(1)
C(13);36(1);37(1);38(1);-9(1);-6(1);6(1)
C(14);38(1);36(1);34(1);-4(1);0(1);-5(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for i146.

;x ;y ;z ;U(eq)

H(4);1877;3967;7097;41
H(5);804;4660;5991;44
H(6);-55;3986;4802;44
H(7);190;2624;4729;39
H(10);2368;669;6432;35
H(11);4076;1450;8281;36
H(12A);1875;167;7782;45
H(12B);1368;804;8467;45
H(12C);2930;196;8625;45
H(13A);3071;2476;8497;44
H(13B);4018;3154;7978;44
H(13C);1989;3228;8184;44
H(14A);1478;1349;4595;43
H(14B);-395;1232;5006;43
H(14C);1103;590;5155;43

Table 6. Torsion angles [deg] for i146.

C(11)#1-O(1)-C(11)-C(1);61.47(14)
C(11)#1-O(1)-C(11)-C(12);-176.39(11)
C(11)-C(1)-C(2)-C(3);176.27(14)
C(11)-C(1)-C(2)-C(13);-4.5(2)
C(2)-C(1)-C(10)-C(9);1.6(2)
C(11)-C(1)-C(10)-C(9);-178.48(14)
C(2)-C(1)-C(11)-O(1);-128.16(15)
C(2)-C(1)-C(11)-C(12);113.17(17)
C(10)-C(1)-C(11)-O(1);51.95(18)
C(10)-C(1)-C(11)-C(12);-66.73(18)
C(10)-C(1)-C(2)-C(13);175.35(14)
C(10)-C(1)-C(2)-C(3);-3.8(2)
C(1)-C(2)-C(3)-C(8);2.8(2)
C(13)-C(2)-C(3)-C(4);4.1(2)
C(1)-C(2)-C(3)-C(4);-176.66(14)
C(13)-C(2)-C(3)-C(8);-176.38(14)
C(8)-C(3)-C(4)-C(5);-1.1(2)
C(2)-C(3)-C(4)-C(5);178.45(15)
C(4)-C(3)-C(8)-C(7);-0.3(2)
C(4)-C(3)-C(8)-C(9);179.97(14)
C(2)-C(3)-C(8)-C(7);-179.83(14)
C(2)-C(3)-C(8)-C(9);0.5(2)
C(3)-C(4)-C(5)-C(6);1.4(2)
C(4)-C(5)-C(6)-C(7);-0.2(2)
C(5)-C(6)-C(7)-C(8);-1.2(2)
C(6)-C(7)-C(8)-C(3);1.4(2)
C(6)-C(7)-C(8)-C(9);-178.88(14)
C(3)-C(8)-C(9)-C(14);177.31(13)
C(7)-C(8)-C(9)-C(10);177.61(14)
C(3)-C(8)-C(9)-C(10);-2.7(2)
C(7)-C(8)-C(9)-C(14);-2.4(2)
C(14)-C(9)-C(10)-C(1);-178.27(14)
C(8)-C(9)-C(10)-C(1);1.7(2)

Symmetry transformations used to generate equivalent atoms:
#1 1/2-x+1,1/2-y,1/2+z+1

Table 7. Hydrogen-bonds for i146 [A and deg.].

D-H...A;d(D-H);d(H...A);d(D...A);<(DHA)