Supporting Information

_N_-(3,3a,4,4a,5,5a,6,6a-Octahydro-1,3-dioxo-4,6-ethenocycloprop[f]isoindol-2-(1_H_)-yl)carboxamides: Identification of Novel Orthopoxvirus Egress Inhibitors.

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General experimental procedure and characterization of final targets. X-ray crystallography data for compound **13**.

General Methods. Melting points were obtained using a MelTemp apparatus, and are uncorrected. Proton NMR spectra were obtained on a Bruker AM 500 MHz spectrometer or a Mercury 300 spectrometer. Elemental analyses for carbon, hydrogen, and nitrogen were performed by Robertson Microlit Laboratories. Analytical thin-layer chromatography (TLC) was conducted on precoated silica gel 60 plates (Whatman). Chromatography was conducted on silica gel 60 under low pressure.

General Procedure for synthesis of compounds 2-14, 16-18. N-(3,3a,4,4a,5,5a,6,6a-octahydro-1,3-dioxo-4,6-ethenocycloprop[f]isoindol-2(1H)-yl)-4-(trifluoromethyl)benzamide (14). A mixture of 2.00 g (9.8 mmol) of 4-(trifluoromethyl) benzoic acid hydrazide, 1.86 g (9.8 mmol) of 4,4a,5,5a,6,6a-hexahydro-4,6-etheno-1H-cycloprop[f]isobenzofuran-1,3(3aH)-dione, and one drop of diisopropylethylamine in 40 mL of absolute ethanol was refluxed for 4.5 h. Upon cooling to rt, 4 mL of water was added, and the product began to crystallize. The suspension was cooled in an ice bath, and the precipitate collected by filtration. The crystalline solid was air-dried affording 3.20 g (87%) of the product as a white solid; Mp 194-195 ºC. ^{1}H NMR, (300 MHz, d^6-DMSO) δ 11.20, 11.09 (2 brs from rotamers, 1H), 8.06 (d, *J*= 7.8 Hz, 2H), 7.90 (d, *J*= 7.8 Hz, 2H), 5.78 (m, 2H), 3.26 (m, 4H), 1.15 (m, 2H), 0.24 (dd, *J*= 7.2, 12.9 Hz, 1H), 0.04 (m, 1H). Anal. calcd. for $C_{19}H_{15}F_3N_2O_3 \bullet 0.25H_2O$: %C, 59.92; %H, 4.10; %F, 14.97; %N, 7.36; %O, 13.65. Found: %C, 59.97; %H, 4.02; %F, 14.94; %N, 7.36; %O, 13.71.

N-(Octahydro-1,3-dioxo-4,6-ethanocycloprop[f]isoindol-2(1H)-yl)-4-(trifluoromethyl)benzamide (15). a) To a solution of 2.50 g (13.1 mmol) of 4,4a,5,5a,6,6a-hexahydro-4,6-etheno-1H-cycloprop[f]isobenzofuran-1,3(3aH)-dione **(1a)** in 25 mL of absolute ethanol in a Paar hydrogenation bottle was added 250 mg (10% by weight) of 10% Pd/C. The mixture was shaken in a Paar hydrogenation apparatus under 50 psi of H_2 for 3 h. The reaction was filtered through celite, and concentrated in vacuo to afford 2.43 g (96%) of the reduced product as a white solid. ^{1}H NMR (500 MHz, d^6-DMSO) δ 3.36 (m, 2H), 2.38 (m, 2H), 1.26 (m, 2H), 1.06 (m, 4H), 0.84 (m, 1H), 0.56 (m, 1H).
b) A solution of 4,4a,5,5a,6,6a-hexahydro-4,6-ethano-1H-cycloprop[f]isobenzofuran-1,3(3aH)-dione (1b; 0.345 g, 1.8 mmol) and 4-(trifluoromethyl) benzoic acid hydrazide (0.366 g, 1.8

mmol) in 11 mL of absolute ethanol was refluxed for 4h. Upon cooling, the reaction was concentrated under vacuum, and the resultant oil subjected to column chromatography (silica gel 60; 1:1 EtOAc/hexanes) affording 0.381g (56% yield) of the product as a white solid. ^{1}H NMR (500 MHz, d^6-DMSO) δ 11.18 (br.s., 1H), 8.05 (d, *J*= 8.5 Hz, 2H), 7.95 (d, *J*= 8.5 Hz, 2H), 3.21 (m, 2H), 2.40 (m, 2H), 1.46 (d, *J*= 8.5 Hz, 2H), 1.10 (m, 4H), 0.82 (m, 1H), 0.53 (m, 1H). Anal. calcd. for $C_{19}H_{17}F_3N_2O_3$: %C, 60.32; %H, 4.53; %N, 7.40. Found: %C, 60.27; %H, 4.37; %N, 7.37.

N-(3,3a,4,4a,5,5a,6,6a-octahydro-1,3-dioxo-4,6-ethenocycloprop[f]isoindol-2(1H)-yl)-4-nitrobenzamide (2). 5% yield. ^{1}H NMR (500 MHz, d^6-DMSO) δ 10.49, 10.23 (2br.s, 1H), 8.38 (m, 2H), 8.12 (m, 2H), 5.80 (m, 2H), 3.30 (m, 4H), 2.90 (s, 6H), 1.18 (m, 2H), 0.27 (m, 1H), 0.08 (m, 1H). Anal. calcd. for $C_{18}H_{15}N_3O_5$: %C, 61.19; %H, 4.28; %N, 11.89. Found %C, 60.97; %H, 4.46; %N, 11.86.

N-(3,3a,4,4a,5,5a,6,6a-octahydro-1,3-dioxo-4,6-ethenocycloprop[f]isoindol-2(1H)-yl)-4-dimethylaminobenzamide (3). 56% yield. ^{1}H NMR (500 MHz, d^6-DMSO) δ 11.14, 10.88 (2br.s, 1H), 7.85 (d, *J*= 7.8 Hz, 2H), 7.55 (d, *J*= 7.8 Hz, 2H), 5.74 (m, 2H), 3.28 (m, 4H), 3.16 (d, *J*= 10 Hz, 6H) 1.10 (m, 2H), 0.20 (m, 1H), 0.02 (m, 1H). Anal. calcd. for $C_{20}H_{21}N_3O_3$: %C, 68.36; %H, 6.02; %N, 11.96. Found: %C, 68.18; %H, 6.01; %N, 11.99.

N-(3,3a,4,4a,5,5a,6,6a-octahydro-1,3-dioxo-4,6-ethenocycloprop[f]isoindol-2(1H)-yl)-4-aminobenzamide (4). 41% yield. ^{1}H NMR (500 MHz, d^6-DMSO) δ 11.49 (br. s, 1H), 7.95 (d, *J*= 7.8 Hz, 2H), 6.56 (d, *J*= 7.8 Hz, 2H), 5.84 (m, 2H), 5.78 (m, 2H), 3.28 (m, 4H), 1.19 (m, 2H), 0.27 (m, 1H), 0.08 (m, 1H). Anal. calcd. for $C_{18}H_{15}N_3O_3$: %C, 66.86; %H, 5.30; %N, 13.00. Found: %C, 66.86; %H, 5.42; %N, 11.80.

N-(3,3a,4,4a,5,5a,6,6a-octahydro-1,3-dioxo-4,6-ethenocycloprop[f]isoindol-2(1H)-yl)-2-pyridinecarboxamide (5). 67% yield. ^{1}H NMR (500 MHz, d^6-DMSO) δ 11.11 (br. s, 1H), 8.70 (d, 4.5 Hz, 1H), 8.04 (m, 4H), 7.69 (m, 2H), 5.76 (m, 2H), 3.27 (m, 4H), 1.17 (m, 2H), 0.26(m, 1H), 0.08 (m, 1H). Anal. calcd. for $C_{17}H_{15}N_3O_3$: %C, 66.01; %H, 4.89; %N, 13.58. Found: %C, 65.91; %H, 4.89; %N, 13.58. Found: %C, 65.91; %H, 5.15; %N, 13.57.

N-(3,3a,4,4a,5,5a,6,6a-octahydro-1,3-dioxo-4,6-ethenocycloprop[f]isoindol-2(1H)-yl)-3-pyridinecarboxamide (6). 62% yield. ^{1}H NMR (500 MHz, d^6-DMSO) δ 11.45 (br. s, 1H), 9.05 (m, 1H), 8.81 (dd, *J*= 1.0, 4.5 Hz, 1H), 8.24 (m, 1H), 7.59 (dd, *J*= 5.0, 8.0 Hz, 1H), 5.79 (m, 2H), 3.36 (m, 4H), 1.19 (m, 2H), 0.27 (m, 1H), 0.07 (m, 1H). Anal. calcd. for $C_{17}H_{15}N_3O_3$: %C, 66.01; %H, 4.89; %N, 13.58. Found: %C, 65.91; %H, 4.89; %N, 13.58. Found: %C, 66.03; %H, 4.73; %N, 13.46.

N-(3,3a,4,4a,5,5a,6,6a-octahydro-1,3-dioxo-4,6-ethenocycloprop[f]isoindol-2(1H)-yl)-4-pyridinecarboxamide (7). 67% yield. ^{1}H NMR (500 MHz, d^6-DMSO) δ 11.40 (v.br. s, 1H), 8.81 (dd, *J*= 1.5, 4.5 Hz, 2H), 7.79 (m, 2H), 5.81 (m, 2H), 3.34 (m, 4H), 1.17 (m, 2H), 0.27 (m, 1H), 0.07 (m, 1H). Anal. calcd. for $C_{17}H_{15}N_3O_3$: %C, 66.01; %H, 4.89; %N, 13.58. Found: %C, 65.91; %H, 4.89; %N, 13.58. Found: %C, 65.94; %H, 4.72; %N, 13.63.

N-(3,3a,4,4a,5,5a,6,6a-octahydro-1,3-dioxo-4,6-ethenocycloprop[f]isoindol-2(1H)-yl)-2-chlorobenzamide (8). 67% yield. ^{1}H NMR (500 MHz, d^6-DMSO) δ 10.78, 10.52 (2br.s., 1H), 7.76 (m, 4H), 5.79 (m, 2H), 3.30 (m, 4H), 1.19 (m, 2H), 0.27 (m, 1H), 0.07 (m, 1H). Anal. calcd. for $C_{18}H_{15}ClN_2O_3$: %C, 63.07; %H, 4.41; %N, 8.17. Found: %C, 63.25; %H, .41; %N, 7.92.

N-(3,3a,4,4a,5,5a,6,6a-octahydro-1,3-dioxo-4,6-ethenocycloprop[f]isoindol-2(1H)-yl)-3-chlorobenzamide (9). 48% yield. ^{1}H NMR (500 MHz, d^6-DMSO) δ 11.14, 10.88 (2br.s., 1H), 7.88 (m, 1H), 7.75 (m, 1H), 7.67 (m, 1H), 7.55 (m, 1H), 5.72 (m, 2H), 3.30 (m, 4H), 1.19 (m, 2H), 0.27 (m, 1H), 0.07 (m, 1H). Anal. calcd. for $C_{18}H_{15}ClN_2O_3$: %C, 63.07; %H, 4.41; %N, 8.17; %Cl, 10.34. Found: %C, 63.06; %H, 4.32; %N, 7.90; %Cl, 10.61.

N-(3,3a,4,4a,5,5a,6,6a-octahydro-1,3-dioxo-4,6-ethenocycloprop[f]isoindol-2(1H)-yl)-4-chlorobenzamide (10). 49% yield. ^{1}H NMR (500 MHz, d^6-DMSO) δ 11.14, 10.88 (2 br.s., 1H), 7.85 (d, *J*= 8.0 Hz, 2H), 7.55 (d, *J*= 8.0 Hz, 2H), 5.72 (m, 2H), 3.30 (m, 4H), 1.19 (m, 2H), 0.27 (m, 1H), 0.07 (m, 1H). Anal. calcd. for $C_{18}H_{15}ClN_2O_3$: %C, 63.07; %H, 4.41; %N, 8.17; %Cl, 10.34. Found: %C, 63.11; %H, 4.61; %N, 7.97; %Cl, 10.56.

N-(3,3a,4,4a,5,5a,6,6a-octahydro-1,3-dioxo-4,6-ethenocycloprop[f]isoindol-2(1H)-yl)-2-bromobenzamide (11). 58% yield. ^{1}H NMR (500 MHz, d^6-DMSO) δ 11.14, 10.88 (2br.s., 1H), 7.85 (d, *J*= 8.0 Hz, 1H), 7.55, (m, 3H), 5.72 (m, 2H), 3.30 (m, 4H), 1.19 (m, 2H), 0.27 (m, 1H), 0.07 (m, 1H). Anal. calcd. for $C_{18}H_{15}BrN_2O_3$: %C, 55.83; %H, 3.90; %N, 7.23, %Br, 20.63. Found: %C, 56.13; %H, 3.83; %N, 6.91; %Br, 20.47.

N-(3,3a,4,4a,5,5a,6,6a-octahydro-1,3-dioxo-4,6-ethenocycloprop[f]isoindol-2(1H)-yl)-3-bromobenzamide (12). 56% yield. ^{1}H NMR (500 MHz, d^6-DMSO) δ 11.14, 10.88 (2br.s., 1H), 8.00 (m, 1H), 7.85 (m, 2H), 7.55 (m, 1H), 5.72 (m, 2H), 3.30 (m, 4H), 1.19 (m, 2H), 0.27 (m, 1H), 0.07 (m, 1H). Anal. calcd. for $C_{18}H_{15}BrN_2O_3$: %C, 55.83; %H, 3.90; %N, 7.23, %Br, 20.63. Found: %C, 55.94; %H, 3.88; %N, 7.02; %Br, 20.29.

N-(3,3a,4,4a,5,5a,6,6a-octahydro-1,3-dioxo-4,6-ethenocycloprop[f]isoindol-2(1H)-yl)-4-bromobenzamide (13). 69% yield. ^{1}H NMR (500 MHz, d^6-DMSO) δ 11.19 (br.s., 1H), 7.83 (m, 2H), 7.76 (d, *J*= 8.5 Hz, 2H), 5.78 (m, 2H), 3.26 (m, 4H), 1.15 (m, 2H), 0.28 (m, 1H), 0.07 (m, 1H). Anal. calcd. for $C_{18}H_{15}BrN_2O_3$: %C, 55.83; %H, 3.90; %N, 7.23, %Br, 20.63. Found: %C, 55.99; %H, 3.88; %N, 7.16; %Br, 20.58.

N-(3,3a,4,4a,5,5a,6,6a-octahydro-1,3-dioxo-4,6-ethenocycloprop[f]isoindol-2(1H)-yl)-4-methoxybenzamide (16). 71% yield. ^{1}H NMR (500 MHz, d^6-DMSO) δ 10.90, 10.65 (2br. s., 1H), 7.81 (m, 2H), 7.06 (m, 2H), 5.80 (m, 2H), 3.84 (s, 3H), 3.28 (m, 4H), 1.18 (m, 2H), 0.27 (m, 1H), 0.07 (m, 1H). Anal. calcd. for $C_{19}H_{18}N_2O_4$: %C, 67.45; %H, 5.36; %N, 8.28. Found: %C, 67.37; %H, 5.50; %N, 8.11.

N-(3,3a,4,4a,5,5a,6,6a-octahydro-1,3-dioxo-4,6-ethenocycloprop[f]isoindol-2(1H)-yl)-1-methyl-1H-pyrrole-2-carboxamide (17). 50% yield. ^{1}H NMR (500 MHz, d^6-DMSO) δ 10.48, 10.20 (2 br.s., 1H), 7.02 (m, 2H), 6.09 (d, *J*= 6.0 Hz, 1H), 5.79 (m, 2H), 3.81 (s, 3H), 3.29 (m, 4H), 1.18 (m, 2H), 0.27 (m, 1H), 0.07 (m, 1H). Anal. calcd. for $C_{17}H_{17}N_3O_3$: %C, 65.58; %H, 5.50; %N, 13.50. Found: %C, 65.47; %H, 5.50; %N, 13.48.

N-(3,3a,4,4a,5,5a,6,6a-octahydro-1,3-dioxo-4,6-ethenocycloprop[f]isoindol-2(1H)-yl)-5-methyl-1H-pyrazole-3-carboxamide(18). 62% yield. ^{1}H NMR (500 MHz, d^6-DMSO) δ 13.12 (br. s., 1H), 10.53 (v.br.s., 1H), 6.44 (s, 1H), 5.77 (m, 2H), 3.27 (m, 2H), 3.12 (m, 2H), 2.32 (s, 3H), 1.18 (m, 2H), 0.26 (m, 1H), 0.07 (m, 1H). Anal. calcd. for $C_{16}H_{16}N_4O_3$: %C, 61.53; %H, 5.16; %N, 17.94. Found: %C, 61.58; %H, 5.23; %N, 18.05.

X-ray crystallography data for compound **13**.

Data collection

A crystal (approximate dimensions 0.41 x 0.29 x 0.23 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary and mounted on a Bruker SMART system for a data collection at 173(2) K. A preliminary set of cell constants was calculated from reflections harvested from three sets of 20 frames. These initial sets of frames were oriented such that orthogonal wedges of reciprocal space were surveyed. This produced initial orientation matrices determined from 114 reflections. The data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 15 seconds and a detector distance of 4.9 cm. A randomly oriented region of reciprocal space was surveyed to the extent of 2.0 hemispheres and to a resolution of 0.77 Å. Three major sections of frames were collected with 0.30º steps in ω at 3 different ϕsettings and a detector position of -28° in 2θ. The intensity data were corrected for absorption and decay (SADABS).[1] Final cell constants were calculated from 3908 strong reflections from the actual data collection after integration (SAINT 6.35A, 2002).[2] Please refer to Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved and refined using SHELXL-V6.12.[3] The space group C2/c was determined based on systematic absences and intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters unless stated otherwise. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to R1 = 0.0381 and wR2 = 0.0967 (F^2, all data).

Structure description

The structure was found to be enantiomeric and a monohydrate. That are three hydrogen bonds

listed in Table 7.

Data collection and structure solution were conducted at the X-Ray Crystallographic Laboratory, 160 Kolthoff Hall, Department of Chemistry, University of Minnesota. All calculations were performed using SGI INDY R4400-SC or Pentium computers using the current SHELXTL suite of programs.

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1 An empirical correction for absorption anisotropy, R. Blessing, Acta Cryst. A51, 33 - 38 (1995).

2 SAINT V6.35A, Bruker Analytical X-Ray Systems, Madison, WI.

3 SHELXTL-Plus V6.12, Bruker Analytical X-Ray Systems, Madison, WI.

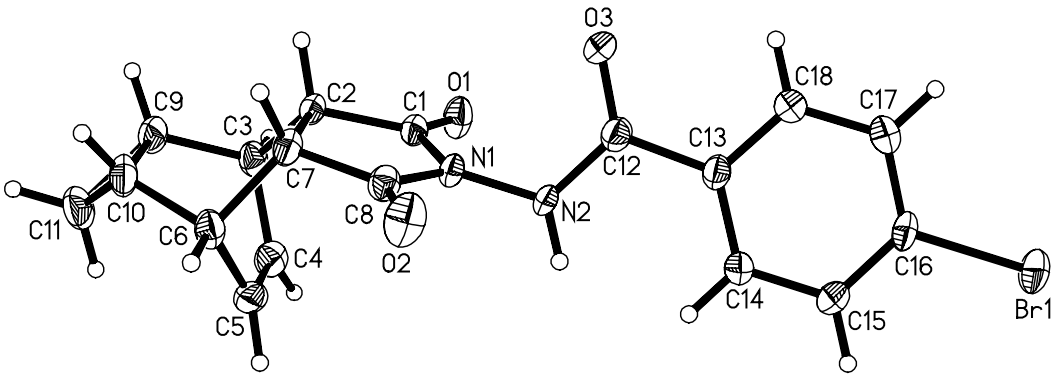

Table 1. Crystal data and structure refinement for compound **13**.

Identification code	compound **13**	
Empirical formula	$C_{18}\ H_{17}\ Br\ N_2\ O_4$	
Formula weight	405.25	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 26.943(3) Å	α= 90°.
	b = 10.7772(13) Å	β= 109.687(2)°.
	c = 12.4623(15) Å	γ = 90°.
Volume	3407.2(7) $Å^3$	
Z	8	
Density (calculated)	1.580 Mg/m^3	
Absorption coefficient	2.439 mm^{-1}	
F(000)	1648	
Crystal habit and color	Colorless, Block	
Crystal size	0.41 x 0.29 x 0.23 mm^3	
Theta range for data collection	1.61 to 27.52°.	
Index ranges	$-34 \le h \le 32$, $0 \le k \le 14$, $0 \le l \le 16$	
Reflections collected	20243	
Independent reflections	3924 [R(int) = 0.0552]	
Observed Reflections	3046	
Completeness to theta = 27.52°	99.9 %	
Absorption correction	Multi-scan	
Max. and min. transmission	0.6039 and 0.4346	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3924 / 1 / 232	
Goodness-of-fit on F^2	1.057	
Final R indices [I>2sigma(I)]	R1 = 0.0381, wR2 = 0.0874	
R indices (all data)	R1 = 0.0569, wR2 = 0.0967	
Largest diff. peak and hole	0.951 and -0.770 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for compound **13.** U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
N1	7863(1)	4721(2)	1700(2)	20(1)
C1	7514(1)	3781(2)	1189(2)	21(1)
O1	7574(1)	2711(2)	1502(2)	30(1)
C2	7071(1)	4341(2)	224(2)	21(1)
C3	6526(1)	4109(2)	374(2)	24(1)
C4	6557(1)	4696(3)	1491(2)	26(1)
C5	6666(1)	5900(3)	1565(2)	26(1)
C6	6733(1)	6456(2)	518(2)	26(1)
C7	7191(1)	5747(2)	282(2)	23(1)
C8	7715(1)	5902(2)	1218(2)	22(1)
O2	7976(1)	6818(2)	1546(2)	36(1)
C9	6137(1)	4801(3)	-633(2)	31(1)
C10	6253(1)	6175(3)	-539(2)	33(1)
C11	5742(1)	5698(3)	-467(3)	41(1)
N2	8309(1)	4514(2)	2632(2)	21(1)
C12	8728(1)	3944(2)	2484(2)	24(1)
O3	8731(1)	3617(2)	1541(2)	37(1)
C13	9186(1)	3700(2)	3544(2)	23(1)
C14	9213(1)	4095(3)	4617(2)	29(1)
C15	9653(1)	3853(3)	5557(2)	29(1)
C16	10065(1)	3211(2)	5416(2)	25(1)
Br1	10674(1)	2896(1)	6696(1)	39(1)
C17	10045(1)	2776(3)	4364(3)	43(1)
C18	9604(1)	3033(3)	3431(2)	40(1)
O4	6905(1)	694(2)	631(2)	29(1)

Table 3. Bond lengths [Å] and angles [°] for compound **13**

N1-N2	1.379(3)	C9-H9A	1.0000
N1-C1	1.383(3)	C10-C11	1.501(4)
N1-C8	1.406(3)	C10-H10A	1.0000
C1-O1	1.210(3)	C11-H11A	0.9900
C1-C2	1.506(3)	C11-H11B	0.9900
C2-C7	1.546(4)	N2-C12	1.352(3)
C2-C3	1.559(4)	N2-H2B	0.8800
C2-H2A	1.0000	C12-O3	1.230(3)
C3-C4	1.506(4)	C12-C13	1.496(3)
C3-C9	1.531(4)	C13-C18	1.382(4)
C3-H3A	1.0000	C13-C14	1.382(4)
C4-C5	1.327(4)	C14-C15	1.383(3)
C4-H4A	0.9500	C14-H14A	0.9500
C5-C6	1.501(4)	C15-C16	1.367(4)
C5-H5A	0.9500	C15-H15A	0.9500
C6-C10	1.534(4)	C16-C17	1.376(4)
C6-C7	1.561(4)	C16-Br1	1.895(2)
C6-H6A	1.0000	C17-C18	1.383(4)
C7-C8	1.509(3)	C17-H17A	0.9500
C7-H7A	1.0000	C18-H18A	0.9500
C8-O2	1.201(3)	O4-H4B	0.80(2)
C9-C11	1.504(4)	O4-H4C	0.80(2)
C9-C10	1.510(4)		

N2-N1-C1	122.2(2)	C7-C2-H2A	110.1
N2-N1-C8	123.2(2)	C3-C2-H2A	110.1
C1-N1-C8	114.5(2)	C4-C3-C9	111.2(2)
O1-C1-N1	123.6(2)	C4-C3-C2	106.4(2)
O1-C1-C2	128.5(2)	C9-C3-C2	103.7(2)
N1-C1-C2	107.9(2)	C4-C3-H3A	111.7
C1-C2-C7	104.97(19)	C9-C3-H3A	111.7
C1-C2-C3	111.4(2)	C2-C3-H3A	111.7
C7-C2-C3	110.0(2)	C5-C4-C3	114.6(2)
C1-C2-H2A	110.1	C5-C4-H4A	122.7

C3-C4-H4A	122.7	C10-C11-C9	60.31(19)
C4-C5-C6	114.8(2)	C10-C11-H11A	117.7
C4-C5-H5A	122.6	C9-C11-H11A	117.7
C6-C5-H5A	122.6	C10-C11-H11B	117.7
C5-C6-C10	110.7(2)	C9-C11-H11B	117.7
C5-C6-C7	107.5(2)	H11A-C11-H11B	114.9
C10-C6-C7	103.5(2)	C12-N2-N1	119.3(2)
C5-C6-H6A	111.6	C12-N2-H2B	120.3
C10-C6-H6A	111.6	N1-N2-H2B	120.3
C7-C6-H6A	111.6	O3-C12-N2	122.3(2)
C8-C7-C2	105.7(2)	O3-C12-C13	121.7(2)
C8-C7-C6	113.2(2)	N2-C12-C13	116.0(2)
C2-C7-C6	108.9(2)	C18-C13-C14	118.7(2)
C8-C7-H7A	109.6	C18-C13-C12	117.4(2)
C2-C7-H7A	109.6	C14-C13-C12	124.0(2)
C6-C7-H7A	109.6	C13-C14-C15	120.8(2)
O2-C8-N1	123.4(2)	C13-C14-H14A	119.6
O2-C8-C7	130.0(2)	C15-C14-H14A	119.6
N1-C8-C7	106.7(2)	C16-C15-C14	119.2(2)
C11-C9-C10	59.7(2)	C16-C15-H15A	120.4
C11-C9-C3	121.4(3)	C14-C15-H15A	120.4
C10-C9-C3	110.3(2)	C15-C16-C17	121.5(2)
C11-C9-H9A	117.2	C15-C16-Br1	119.5(2)
C10-C9-H9A	117.2	C17-C16-Br1	119.0(2)
C3-C9-H9A	117.2	C16-C17-C18	118.6(3)
C11-C10-C9	59.95(19)	C16-C17-H17A	120.7
C11-C10-C6	122.8(3)	C18-C17-H17A	120.7
C9-C10-C6	110.7(2)	C13-C18-C17	121.2(3)
C11-C10-H10A	116.6	C13-C18-H18A	119.4
C9-C10-H10A	116.6	C17-C18-H18A	119.4
C6-C10-H10A	116.6	H4B-O4-H4C	104(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for compound **13**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U_{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N1	15(1)	23(1)	17(1)	0(1)	0(1)	0(1)
C1	17(1)	23(1)	20(1)	-2(1)	3(1)	1(1)
O1	26(1)	22(1)	32(1)	3(1)	-2(1)	2(1)
C2	18(1)	23(1)	17(1)	-1(1)	0(1)	2(1)
C3	16(1)	24(1)	28(1)	-2(1)	3(1)	-1(1)
C4	19(1)	34(2)	26(1)	1(1)	8(1)	-4(1)
C5	20(1)	33(1)	23(1)	-8(1)	4(1)	-1(1)
C6	22(1)	21(1)	30(1)	0(1)	3(1)	4(1)
C7	20(1)	28(1)	19(1)	5(1)	3(1)	1(1)
C8	21(1)	25(1)	21(1)	4(1)	6(1)	0(1)
O2	32(1)	26(1)	41(1)	5(1)	2(1)	-10(1)
C9	21(1)	36(2)	26(1)	-7(1)	-4(1)	5(1)
C10	27(1)	33(2)	29(1)	2(1)	-1(1)	11(1)
C11	21(1)	47(2)	44(2)	-11(2)	-3(1)	11(1)
N2	15(1)	31(1)	13(1)	-2(1)	-1(1)	2(1)
C12	19(1)	30(1)	20(1)	-1(1)	4(1)	-1(1)
O3	27(1)	63(1)	17(1)	-5(1)	3(1)	11(1)
C13	16(1)	31(1)	20(1)	1(1)	3(1)	1(1)
C14	22(1)	42(2)	20(1)	-1(1)	4(1)	10(1)
C15	26(1)	41(2)	17(1)	0(1)	4(1)	5(1)
C16	18(1)	31(1)	21(1)	4(1)	-2(1)	3(1)
Br1	30(1)	51(1)	26(1)	4(1)	-5(1)	16(1)
C17	26(2)	67(2)	31(2)	-7(2)	4(1)	20(2)
C18	26(2)	66(2)	24(1)	-9(1)	5(1)	15(1)
O4	30(1)	34(1)	18(1)	2(1)	2(1)	-9(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for compound **13**.

	x	y	z	U(eq)
H2A	7070	3994	-521	25
H3A	6442	3204	357	29
H4A	6502	4241	2095	32
H5A	6700	6373	2229	31
H6A	6805	7368	610	31
H7A	7219	6030	-459	28
H9A	6052	4415	-1404	37
H10A	6235	6614	-1258	39
H11A	5417	5854	-1122	49
H11B	5694	5713	287	49
H2B	8320	4749	3315	25
H14A	8925	4538	4709	34
H15A	9670	4130	6292	34
H17A	10329	2309	4281	52
H18A	9587	2746	2699	48
H4B	7072(12)	1300(20)	890(30)	35
H4C	6728(11)	890(30)	0(20)	35

Table 6. Torsion angles [°] for compound **13**.

N2-N1-C1-O1	-1.1(4)	C2-C3-C9-C11	-129.7(3)
C8-N1-C1-O1	-178.8(2)	C4-C3-C9-C10	50.5(3)
N2-N1-C1-C2	178.6(2)	C2-C3-C9-C10	-63.4(3)
C8-N1-C1-C2	0.9(3)	C3-C9-C10-C11	-115.3(3)
O1-C1-C2-C7	176.6(3)	C11-C9-C10-C6	116.8(3)
N1-C1-C2-C7	-3.1(3)	C3-C9-C10-C6	1.5(3)
O1-C1-C2-C3	57.6(3)	C5-C6-C10-C11	14.2(4)
N1-C1-C2-C3	-122.1(2)	C7-C6-C10-C11	129.2(3)
C1-C2-C3-C4	59.4(3)	C5-C6-C10-C9	-52.6(3)
C7-C2-C3-C4	-56.6(3)	C7-C6-C10-C9	62.3(3)
C1-C2-C3-C9	176.8(2)	C6-C10-C11-C9	-96.4(3)
C7-C2-C3-C9	60.8(2)	C3-C9-C11-C10	96.6(3)
C9-C3-C4-C5	-54.2(3)	C1-N1-N2-C12	75.4(3)
C2-C3-C4-C5	58.0(3)	C8-N1-N2-C12	-107.1(3)
C3-C4-C5-C6	0.7(3)	N1-N2-C12-O3	1.1(4)
C4-C5-C6-C10	53.5(3)	N1-N2-C12-C13	-177.2(2)
C4-C5-C6-C7	-58.8(3)	O3-C12-C13-C18	-3.3(4)
C1-C2-C7-C8	4.1(3)	N2-C12-C13-C18	175.1(3)
C3-C2-C7-C8	124.0(2)	O3-C12-C13-C14	177.0(3)
C1-C2-C7-C6	-117.9(2)	N2-C12-C13-C14	-4.7(4)
C3-C2-C7-C6	2.1(3)	C18-C13-C14-C15	1.4(4)
C5-C6-C7-C8	-63.5(3)	C12-C13-C14-C15	-178.9(3)
C10-C6-C7-C8	179.3(2)	C13-C14-C15-C16	-0.2(4)
C5-C6-C7-C2	53.8(3)	C14-C15-C16-C17	-1.5(5)
C10-C6-C7-C2	-63.4(2)	C14-C15-C16-Br1	179.1(2)
N2-N1-C8-O2	3.3(4)	C15-C16-C17-C18	1.9(5)
C1-N1-C8-O2	-179.0(3)	Br1-C16-C17-C18	-178.7(3)
N2-N1-C8-C7	-175.9(2)	C14-C13-C18-C17	-0.9(5)
C1-N1-C8-C7	1.8(3)	C12-C13-C18-C17	179.3(3)
C2-C7-C8-O2	177.2(3)	C16-C17-C18-C13	-0.7(5)
C6-C7-C8-O2	-63.7(4)		
C2-C7-C8-N1	-3.6(3)		
C6-C7-C8-N1	115.5(2)		
C4-C3-C9-C11	-15.7(4)		

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for compound **13** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N2-H2B...O4#1	0.88	1.91	2.735(3)	154.5
O4-H4B...O1	0.80(2)	2.00(2)	2.799(3)	172(3)
O4-H4C...O3#2	0.80(2)	1.97(2)	2.772(3)	178(3)

Symmetry transformations used to generate equivalent atoms:

#1 -x+3/2,y+1/2,-z+1/2 #2 -x+3/2,-y+1/2,-z

Unit cell for compound 13.