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Metalloprobes. Monitoring Impact of Geometric Isomers on Accumulation Profiles in Rat Cardiomyoblast and Human Breast Carcinoma Cells

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Experimental Section.

General Methods.

All reagents were purchased from Sigma-Aldrich unless otherwise stated. The linear tetramine, 1,2-ethylenediaminobis(2,2-dimethylaminopropane) was synthesized as described previously[1,2]. ^{1}H NMR and proton-decoupled ^{13}C NMR spectra were recorded on Bruker (400 MHz) spectrometer; chemical shifts are reported in δ (ppm) with reference to TMS. Mass spectra were obtained from the Washington University Resource for Biomedical and Bioorganic Mass Spectrometry using samples diluted in 50/50 methanol/water containing 0.1% formic acid and analyzed via HRESI and University of Missouri Mass Spectrometry facility using nitrobenzyl alcohol (NBA) as matrix and analyzed via HRFab. Electrostatic potential maps, dipole moment and polarizabilities were calculated using Spartan 10 (Wave Function, Inc., Irvine, CA) with PM6, Geometry Optimization (Analytical Gradient). HPLC analysis was performed with a Waters System 600 equipped with dual l-detector 2487 (set to 280 and 214 nm) and a c-detector (Bioscan) for identification of radiopeaks. Gallium(III) complex **5** and **7** and its gallium-67 labeled analogues were assessed for purity on a C-18 reversed-phase column (Vydac TP, 10 mm, 300 A°) using an eluent gradient of ethanol and saline (isocratic 20% ethanol in saline for 5 min; gradient from 20% to 90% ethanol in saline from 5–40 min, at a flow of 2 mL/min). Radiochemical purity was determined on C-18 plates employing a mobile eluent mixture of 90% ethanol in saline, using a radio-TLC (Bioscan).

General procedure for the selective ortho formylation of 2-isopropoxyphenol 1a and 2-isopropylpheonl 1c. Substituted 2-hydroxy benzaldehyde derivatives were obtained by following methodology described previously [2]. Briefly, 2-Isopropoxy/2-Isopropyl phenol (1 mmol) was treated with five equivalents of anhydrous magnesium chloride (5 mmol), and ten equivalent of anhydrous triethylamine (10 mmol) were suspended in anhydrous acetonitrile (50 mL), and the suspension stirred for 1 h at room temperature. Then, five equivalent of *p*-formaldehyde (5 mmol) was added to the mixture and the contents were heated at reflux for 4 h. The reaction mixture was cooled to room temperature, hydrolyzed, acidified with 10% HCl (50 mL), and extracted with ether

(3 × 200 mL). The combined organic extract was dried over anhydrous sodium sulfate, filtered, concentrated, and the residue was purified on silica gel GF254 (Analtech, USA) using a hexane/ethyl acetate (70/30) eluent mixture.

2-Hydroxy-3-isopropoxybenzaldehyde 3a. Following the above general procedure, 2-isopropoxyphenol **1a** (1.34 mmol), anhydrous magnesium chloride (6.73 mmol), anhydrous triethylamine (13.4 mmol), and *p*-formaldehyde (6.72 mmol) resulted **3a**, 57% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.39 (d, 6H), 4.59 (quintet, 1H), 6.94 (t, 1H), 7.13 – 7.21 (dd, 2H), 9.92 (s, 1H), 10.98 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 22.0, 72.0, 119.5, 121.2, 122.6, 125.2, 146.4, 152.9, 196.5; MS(HRESI) Calcd for $[C_{10}H_{12}O_3\text{-}H2O+H]^+$: 163.0754; found: 163.0759.

2-Hydroxy-3-isopropylbenzaldehyde 3b. Following the above general procedure, 2-isopropylphenol **1b** (7.35 mmol), anhydrous magnesium chloride (36.76 mmol), anhydrous triethylamine (73.5 mmol), and *p*-formaldehyde (36.76 mmol) resulted **3b**, 64% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.24 (d, 6H), 3.37 (quintet, 1H), 6.97 (t, 1H), 7.36-7.38 (dd, 1H), 7.45-7.47 (dd, 1H), 9.86 (s, 1H), 11.39 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 22.2, 26.2, 119.6, 120.1, 131.3, 133.6, 137.0, 159.2, 196.9; MS(HREI) Calcd for $[C_{10}H_{12}O_2]^+$: 164.0837; found: 164.0836.

Selective mono-O-alkylation of 2,4-dihydroxybenzaldehyde 2. 2,4-dihydroxybenzaldehyde **2** (7.25 mmol) and potassium carbonate (21.74 mmol) dissolved in anhydrous DMF (10 mL) were heated to 100 °C for 30 min, reacted with the drop-wise addition of 2-bromopropane (7.97 mmol) at room temperature, and contents were stirred under argon atmosphere for 15 h at room temperature. Following the reaction, the contents were diluted with water (100 mL), the product extracted with ether (3 x 200 mL), combined ether extract was washed with water (2 x 100 mL), dried over anhydrous sodium sulfate, filtered, concentrated, and purified on silica gel GF254 (Analtech, USA) using hexanes/ethyl acetate (70/30) as an eluent mixture to give pure **3c**, 80% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.37 (d, 6H), 4.62 (quintet, 1H), 6.40 (d, 1H), 6.48-6.51 (dd, 1H), 7.40 (d, 1H), 9.69 (s, 1H), 11.48 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 21.8, 70.6, 101.7, 109.4, 114.8, 135.3, 164.5, 165.3, 194.2; MS(HREI) Calcd for $[C_{10}H_{12}O_3]^+$: 180.0786; found: 180.0791.

General procedure for the synthesis of ligand 4 and 7. Isopropoxy/isopropyl substituted salcyladehyde **3** (3 mmol) and N,N'-bis(2,2-dimethyl-3-aminopropyl)-ethylenediamine or triethylenetetramine (1 mmol) were dissolved in ethanol (10 mL), refluxed for 45 min, and purified by methods described previously.[1-4]

General procedure for the synthesis of metalloprobes. All precursor ligands were obtained using previously published precedures.[1-4] Briefly, the ligand (0.2 mmol) dissolved in methanol (5 mL) was treated with dropwise addition of gallium(III) acetylacetonate (0.2 mmol) dissolved in methanol. The contents were refluxed for 3 h. Then, potassium iodide (0.2 mmol) dissolved in hot water (0.5 mL) was added and reaction mixture was refluxed for 15 min, brought to room temperature slowly, and slow evaporation over several days yielded crystalline material.

(1,2-Ethylenediamino-bis[2,2-dimethyl-3-{(3-isopropoxyphenyl-2-ate)methyleneamino}-propane]) gallium(III)iodide 8a. Following the above general procedure, the ligand **7a** (100 mg, 0.18 mmol), gallium(III) acetylacetonate (66.2 mg, 0.18 mmol), and potassium iodide (30 mg, 0.18 mmol) resulted **8a**. 30 % yield. ^{1}H NMR (400 MHz, Methanol-d_4) δ: 0.88 (s, 6H), 1.03 (s, 6H), 1.35-1.37 (dd, 12H), 2.75 (t, 4H), 2.97 (d, 2H) 3.07 (d, 2H), 3.80 (d, 2H), 3.93 (d, 2H), 4.67 (quintet, 2H), 6.66 (t, 2H), 6.86-6.89 (dd, 2H), 7.04-7.07 (dd, 2H), 8.07 (s, 2H); ^{13}C NMR (100 MHz, Methanol-d_4) δ: 16.9, 21.1, 21.3, 21.4, 25.5, 35.6, 56.9, 59.6, 69.4, 70.3, 70.3, 115.9, 119.6, 120.6, 125.9, 149.0, 158.5, 170.2. MS(HRESI) Calcd for $[C_{32}H_{48}N_4O_4Ga]^+$: 621.2926; found: m/z = 621.2930 and Calcd for $[^{13}C_{32}H_{48}N_4O_4Ga]^+$: 622.2959; found: m/z = 622.2967; log P: 0.98.

(1,2-Ethylenediamino-bis[2,2-dimethyl-3-{(3-isopropylphenyl-2-ate)methyleneamino}-propane]) gallium(III)iodide 8b. Following the above general procedure, ligand **7b** (153 mg, 0.26 mmol), gallium(III) acetylacetonate (94 mg, 0.26 mmol), and KI (44 mg) resulted in **8b**, 73% yield. ^{1}H NMR (400 MHz, methanol-d_4) δ: 0.86 (s, 6H), 0.99 (s, 6H), 1.22 (d, 6H), 1.35 (d, 6H), 2.80 (d, 2H), 2.88 (d, 2H) 2.97 (d, 2H), 3.15 (d, 2H), 3.48-3.53 (dd, 2H), 3.60-3.70 (m, 4H), 4.45 (br, s, 1H), 6.71 (t, 2H), 7.10-7.12 (dd, 2H), 7.37-7.39 (dd, 2H), 8.08 (s, 2H); ^{13}C

NMR (100 MHz, methanol-d_4) δ: 20.97, 21.6, 23.2, 25.5, 25.9, 29.4, 35.7, 47.9, 60.1, 60.3, 68.8, 116.3, 118.7, 131.3, 132.0, 139.6, 164.2, 170.4 MS(HRFAB) Calcd for $[C_{32}H_{48}N_4O_2Ga]^+$: 589.3033, found: m/z= 589.3049; log *P*: 1.0.

(1,2-Ethylenediamino-bis[2,2-dimethyl-3-{(4-isopropoxyphenyl-2-ate)methyleneamino}-propane]) gallium(III)iodide 8c. Following the above general procedure, ligand **7c** (153 mg, 0.26 mmol), gallium(III) acetylacetonate (94 mg, 0.26 mmol), and KI (44 mg) resulted in **8c**, 61% yield. ^{1}H NMR (400 MHz, methanol-d_4) δ: 0.88 (s, 6H), 1.00 (s, 6H), 1.33 (d, 12H), 2.77 (d, 2H), 2.92-3.03 (m, 4H) 3.49 (d, 2H), 3.99 (d, 2H), 4.66 (quintet, 2H) 6.28-6.33 (m, 4H), 7.12 (d, 2H), 7.91 (s, 2H); ^{13}C NMR (100 MHz, methanol-d_4) δ: 20.7, 20.9, 21.3, 25.7, 35.6, 60.3, 69.3, 69.6, 105.3, 105.4, 107.0, 113.2, 135.5, 164.3, 168.7, 169.9. MS(HRFAB) Calcd for $[C_{32}H_{48}N_4O_4Ga]^+$: 621.2931, found: m/z= 621.2943.

(1,2-Ethylenediamino-bis[1-{(3-isopropoxyphenyl-2-ate)methyleneamino}-ethane]) gallium(III)iodide 5a. Following the above general procedure, ligand **4a** (153 mg, 0.26 mmol), gallium(III) acetylacetonate (94 mg, 0.26 mmol), and KI (44 mg) resulted in **5a**, 42% yield. ^{1}H NMR (400 MHz, methanol-d_4) δ: 0.80 (s, 6H), 1.07 (s, 6H), 2.96 (d, 2H), 3.04-3.09 (dd, 2H), 3.23-3.29 (m, 2H), 3.55-3.63 (m, 2H), 3.84-3.97 (m, 2H), 4.05-4.13 (m, 1H), 6.54-6.59 (dt, 2H), 6.91-6.93 (dd, 2H), 7.03-7.05 (dd, 2H), 8.64 (s, 2H); ^{13}C NMR (100 MHz, methanol-d_4) δ: 20.4, 21.6, 44.7, 45.9, 51.3, 72.6, 114.9, 119.6, 126.5, 129.1, 148.8, 160.5, 170.8. MS(HRFAB) Calcd for $[C_{26}H_{36}N_4O_4Ga]^+$: 537.1992, found: m/z= 537.1986; log *P*: 1.25.

(1,2-Ethylenediamino-bis[1-{(3-isopropylphenyl-2-ate)methyleneamino}-ethane]) gallium(III)iodide 5b. Following the above general procedure, ligand **4b** (153 mg, 0.26 mmol), gallium(III) acetylacetonate (94 mg, 0.26 mmol), and KI (44 mg) resulted in **5b**, 59% yield. ^{1}H NMR (400 MHz, methanol-d_4) δ: 0.77 (s, 6H), 1.05 (s, 6H), 2.91 (d, 2H), 3.02-3.07 (m, 2H) 3.14 (quintet, 2H), 3.21 (d, 2H), 3.49-3.57 (m, 2H), 3.84-3.89 (m, 2H), 4.02-4.10 (m, 2H), 6.58 (t, 2H), 7.08-7.15 (dd, 4H), 8.58 (s, 2H); ^{13}C NMR (100 MHz, methanol-d_4) δ: 21.1, 21.5, 26.4, 44.0, 45.6, 50.9, 53.4, 115.1, 117.6, 130.9, 131.9, 140.0, 165.4, 170.9. MS(HRFAB) Calcd for $[C_{26}H_{36}N_4O_2Ga]$+ : 505.2094, found: m/z= 505.2101; log *P*: 0.2.

(1,2-Ethylenediamino-bis[1-{(4-isopropoxyphenyl-2-ate)methyleneamino}-ethane]) gallium(III)iodide 5c. Following the above general procedure, ligand **4c** (153 mg, 0.26 mmol), gallium(III) acetylacetonate (94 mg, 0.26 mmol), and KI (44 mg) resulted in **5c**, 54% yield. ^{1}H NMR (400 MHz, methanol-d_4) δ: 1.25-1.28 (m, 6H), 2.75 (d, 1H), 2.87-2.92 (m, 1H), 3.08 (d, 1H) 3.36-3.44 (m, 1H), 4.57 (quintet, 1H), 6.20 (d, 1H), 6.29-6.31 (dd, 1H), 7.21 (d, 1H), 8.44 (s, 1H); ^{13}C NMR (100 MHz, methanol-d_4) δ: 20.8, 20.9, 43.2, 44.4, 49.7, 69.5, 69.6, 105.0, 105.1, 106.7, 112.4, 136.3, 164.5, 169.2, 169.6. MS(HRFAB) Calcd for $[C_{26}H_{36}N_4O_4Ga]$+ : 537.1992, found: m/z= 537.1983; Log *P*: 0.3.

Radiochemistry

Preparation of ^{67}Ga-Metalloprobes 6a-c, 9a-c: Radiolabeled ^{67}Ga-metalloprobes were synthesized by following a procedure described earlier[1] with slight modifications. Briefly, ^{67}Ga was obtained as a commercial citrate salt in water (Nordion, Canada), converted into chloride using HCl (6 N), extracted in ether (2 × 2 mL), and evaporated, and the residue was converted into ^{67}Ga(acetylacetonate)$_3$ by reacting with acetylacetone using standard procedures. The radiolabeled ^{67}Ga-metalloprobes were obtained through a ligand exchange reaction involving ^{67}Ga(acetylacetonate)3 and heptadentate Schiff-base precursor ligands **4a-c**, **7a-c** dissolved in ethanol, heated at 100 °C for 40 min. The reaction was followed using thin-layer chromatography plates (C-18) employing a radiometric scanner (Bioscan), using an eluent mixture of 90/10 ethanol/saline (Rf: 0.80). Finally, ^{67}Ga-Galmydar was purified by radio-HPLC on a C-18 reversed-phase column (Vydac TP, 10 µm, 300 Å), using the gradient eluent mixture of ethanol and saline described above. The fractions were collected, concentrated, and employed for bioassays.

X-ray Crystallography.

Crystals suitable for X-ray crystallography were grown by dissolving **5a** in refluxing methanol, slowly bringing the solution to room temperature, and then slow evaporation of the methanol solution overnight. A single crystal with approximate dimensions 0.271 x 0.262 x 0.214 mm^3 was mounted on a glass fiber in a random orientation. Preliminary examination and data collection were performed using a Bruker Kappa Apex II (Charge Coupled Device

(CCD) Detector system) single crystal X-Ray diffractometer, equipped with an Oxford Cryostream LT device. All data were collected using graphite monochromated Mo Kα radiation (λ = 0.71073 Å) from a fine focus sealed tube X-Ray source. Preliminary unit cell constants were determined with a set of 36 narrow frame scans. The collected data set consisted of combinations of ϖ and ϕ scan frames with a scan width of 0.5° and counting time of 15 seconds/frame at a crystal to detector distance of 4.0 cm. The collected frames were integrated using an orientation matrix determined from the narrow frame scans. Apex II and SAINT software packages were used for data collection and data integration. Analysis of the integrated data did not show any decay. Final cell constants were determined by global refinement of xyz centroids of 13630 reflections from the complete data set. Collected data were corrected for systematic errors using SADABS [5] based on the Laue symmetry using equivalent reflections. Structure solution and refinement were carried out using the SHELXTL- PLUS software package. The structure was solved by direct methods and refined successfully in the orthorhombic space group, Pbca. Full matrix least-squares refinement was carried out by minimizing $\Sigma w(F_o^2-F_c^2)^2$. The non-hydrogen atoms were refined anisotropically to convergence. The N-H hydrogens were located and refined with geometrical restraints. Other hydrogen atoms were treated using appropriate riding model (AFIX m3). X-ray crystallographic data for **5a** (crystal data and structure refinement parameters, atomic coordinates, inter-atomic distances and angles, anisotropic displacement parameters, hydrogen coordinates, and torsion angles) are included in **Tables 1-7**.

BIOASSAYS

Cell Culture.

Rat cardiomyoblasts (H9c2(2-1)) and human breast carcinoma (MCF-7) cells were grown in DMEM (high glucose) supplemented with L-glutamine (2 mM) and heat-inactivated fetal calf serum (10%). [2]

Cell Transport Studies.

Cellular transport studies for **6** and **9** were performed in 24-well tissue culture treated plates. Cells (100,000/well) were plated in media and allowed to recover overnight. Media was removed from cells and replaced with media

containing the desired concentrations of **6** and **9 (**74 kBq/mL; Approx. Specific Activity: 16 Ci/mol**)**. Cells were allowed to incubate under normal incubation conditions (37°C, 5% CO_2 atmosphere) for 90min, and then washed 3x with 4°C DPBS (Dulbecco's phosphate buffered saline lacking $CaCl_2$ and $MgCl_2$). Cells were then extracted in 1% sodium dodecyl sulfate with 10 mM sodium borate. Aliquots of the loading solution and ^{67}Ga-complexes **6** and **9** stock solutions also were obtained for standardizing cellular data with the extracellular concentration of **6** and **9**. All cell extracts, ^{67}Ga-complexes **6** and **9** stock solutions, and loading solution samples were assayed for γ-activity in a well-type sodium iodide γ-counter (Cobra II; Packard). Protein mass was estimated by the bicinchoninic acid (BCA) analysis (Pierce Chemical Co.), using bovine serum albumin as the protein standard. Data are reported as fmol ^{67}Ga-complexes **6** and **9** $(nM_0)^{-1}$ (mg protein)$^{-1}$ as previously described,[1,6] with nM_0 representing the total concentration of ^{67}Ga-complexes **6** and **9** in the extracellular buffer.

Fluorescence Microscopic Studies.

For microscopy, H9c2(2-1) cells were plated onto borosilicate 8-well chambered (25,000 cells/chamber) slides (Labtek) and allowed to grow to approximately 70% confluence at 37°C under 5% CO_2 atmosphere. Prior to imaging, cells were incubated with **5a** and **8a** (20 μM in imaging media containing 0.1% ethanol) for 30 minutes at 37°C under a continuous influx of 5% CO_2 atmosphere. Cellular accumulation of the metalloprobe was assessed using a Nikon Ti-E PFS inverted microscope equipped with a Nikon 60x 0.3 NA Plan APO objective (oil), Prior H117 ProScan flat top linear encoded stage, and Prior Lumen 200PRO illumination system with standard DAPI and FITC filter sets. Images were acquired using a Photometrics CoolSNAP HQ2 digital camera and MetaMorph Microscopy and Imaging Analysis Software (version 7.7.0.0, Molecular Devices). Images were processed and analyzed using the ImageJ software package (NIH) [6,7].

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Table 1. Crystal data and structure refinement for **5a**.

Identification code	**5a/**v4515/lt/smart/JOT-II-5(170-21)	
Empirical formula	C_{26} H_{36} Ga I N_4 O_4	
Formula weight	665.21	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 11.2699(9) Å	α= 90°.
	b = 17.6804(14) Å	β= 109.088(3)°.
	c = 14.9326(12) Å	γ = 90°.
Volume	2811.8(4) $Å^3$	
Z	4	
Density (calculated)	1.571 Mg/m^3	
Absorption coefficient	2.114 mm^{-1}	
F(000)	1344	
Crystal size	0.271 x 0.262 x 0.214 mm^3	
Theta range for data collection	1.846 to 36.355°.	
Index ranges	-18≤h≤18, -29≤k≤29, -24≤l≤24	
Reflections collected	90565	
Independent reflections	13630 [R(int) = 0.0406]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7471 and 0.6882	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	13630 / 6 / 350	
Goodness-of-fit on F^2	1.029	
Final R indices [I>2sigma(I)]	R1 = 0.0228, wR2 = 0.0505	
R indices (all data)	R1 = 0.0309, wR2 = 0.0534	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.664 and -0.375 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **5a**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
I(1)	1875(1)	4301(1)	3574(1)	22(1)
I(1')	1874(1)	4320(1)	3549(1)	11(1)
Ga(1)	2304(1)	6864(1)	4617(1)	10(1)
O(1)	3290(1)	7765(1)	4784(1)	12(1)
O(2)	5184(1)	8767(1)	4943(1)	16(1)
O(3)	2399(1)	6568(1)	3408(1)	13(1)
O(4)	2269(1)	6487(1)	1565(1)	14(1)
N(1)	699(1)	7477(1)	3998(1)	13(1)
N(2)	3832(1)	6227(1)	5283(1)	14(1)
N(3)	2243(1)	6954(1)	6012(1)	15(1)
N(4)	972(1)	5998(1)	4504(1)	15(1)
C(1)	2995(1)	8406(1)	4321(1)	11(1)
C(2)	3978(1)	8933(1)	4380(1)	13(1)
C(3)	3726(1)	9636(1)	3955(1)	16(1)
C(4)	2491(1)	9850(1)	3446(1)	18(1)
C(5)	1520(1)	9352(1)	3357(1)	15(1)
C(6)	1754(1)	8626(1)	3780(1)	12(1)
C(7)	673(1)	8152(1)	3661(1)	14(1)
C(8)	-480(1)	7064(1)	3837(1)	21(1)
C(9)	-212(1)	6221(1)	3762(1)	20(1)
C(10)	883(1)	5827(1)	5452(1)	24(1)
C(11)	1147(1)	6528(1)	6088(1)	24(1)
C(12)	3488(1)	6728(1)	6684(1)	21(1)
C(13)	4040(1)	6070(1)	6287(1)	21(1)
C(14)	4690(1)	6061(1)	4918(1)	15(1)
C(15)	4576(1)	6167(1)	3934(1)	13(1)
C(16)	3423(1)	6370(1)	3233(1)	11(1)
C(17)	3393(1)	6338(1)	2273(1)	13(1)
C(18)	4462(1)	6211(1)	2038(1)	18(1)
C(19)	5614(1)	6070(1)	2747(1)	22(1)
C(20)	5656(1)	6029(1)	3678(1)	19(1)

C(21)	5780(1)	8131(1)	4642(1)	20(1)
C(22)	6564(4)	7800(3)	5591(3)	26(1)
C(23)	6537(4)	8349(3)	4017(3)	26(1)
C(22')	6729(5)	7748(4)	5474(4)	26(1)
C(23')	6394(5)	8494(3)	3965(4)	26(1)
C(24)	1310(1)	5907(1)	1475(1)	15(1)
C(25)	1582(1)	5232(1)	949(1)	23(1)
C(26)	64(1)	6283(1)	976(1)	23(1)

Table 3. Bond lengths [Å] and angles [°] for **5a**.

Ga(1)-O(1)	1.9122(7)
Ga(1)-O(3)	1.9154(7)
Ga(1)-N(2)	2.0256(9)
Ga(1)-N(1)	2.0521(9)
Ga(1)-N(4)	2.1116(9)
Ga(1)-N(3)	2.1132(9)
O(1)-C(1)	1.3126(12)
O(2)-C(2)	1.3758(12)
O(2)-C(21)	1.4551(14)
O(3)-C(16)	1.3119(12)
O(4)-C(17)	1.3840(12)
O(4)-C(24)	1.4632(13)
N(1)-C(7)	1.2917(13)
N(1)-C(8)	1.4660(14)
N(2)-C(14)	1.2893(14)
N(2)-C(13)	1.4660(14)
N(3)-C(11)	1.4822(15)
N(3)-C(12)	1.4877(15)
N(3)-H(3)	0.846(15)
N(4)-C(9)	1.4818(15)
N(4)-C(10)	1.4822(15)
N(4)-H(4)	0.871(15)
C(1)-C(6)	1.4212(14)
C(1)-C(2)	1.4279(14)
C(2)-C(3)	1.3820(14)
C(3)-C(4)	1.4028(16)
C(3)-H(3A)	0.9500
C(4)-C(5)	1.3773(16)
C(4)-H(4A)	0.9500
C(5)-C(6)	1.4171(14)
C(5)-H(5)	0.9500
C(6)-C(7)	1.4401(14)
C(7)-H(7)	0.9500
C(8)-C(9)	1.5326(16)

C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(9)-H(9A)	0.9900
C(9)-H(9B)	0.9900
C(10)-C(11)	1.5303(16)
C(10)-H(10A)	0.9900
C(10)-H(10B)	0.9900
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900
C(12)-C(13)	1.5270(16)
C(12)-H(12A)	0.9900
C(12)-H(12B)	0.9900
C(13)-H(13A)	0.9900
C(13)-H(13B)	0.9900
C(14)-C(15)	1.4450(15)
C(14)-H(14)	0.9500
C(15)-C(20)	1.4107(15)
C(15)-C(16)	1.4226(14)
C(16)-C(17)	1.4232(14)
C(17)-C(18)	1.3789(15)
C(18)-C(19)	1.4035(16)
C(18)-H(18)	0.9500
C(19)-C(20)	1.3778(17)
C(19)-H(19)	0.9500
C(20)-H(20)	0.9500
C(21)-C(23)	1.505(4)
C(21)-C(22')	1.510(4)
C(21)-C(22)	1.521(4)
C(21)-C(23')	1.539(5)
C(21)-H(21)	1.0000
C(22)-H(22A)	0.9800
C(22)-H(22B)	0.9800
C(22)-H(22C)	0.9800
C(23)-H(23A)	0.9800
C(23)-H(23B)	0.9800
C(23)-H(23C)	0.9800

C(22')-H(22D)	0.9800
C(22')-H(22E)	0.9800
C(22')-H(22F)	0.9800
C(23')-H(23D)	0.9800
C(23')-H(23E)	0.9800
C(23')-H(23F)	0.9800
C(24)-C(26)	1.5117(16)
C(24)-C(25)	1.5136(16)
C(24)-H(24)	1.0000
C(25)-H(25A)	0.9800
C(25)-H(25B)	0.9800
C(25)-H(25C)	0.9800
C(26)-H(26A)	0.9800
C(26)-H(26B)	0.9800
C(26)-H(26C)	0.9800

O(1)-Ga(1)-O(3)	98.16(3)
O(1)-Ga(1)-N(2)	92.01(3)
O(3)-Ga(1)-N(2)	90.62(3)
O(1)-Ga(1)-N(1)	90.42(3)
O(3)-Ga(1)-N(1)	91.88(3)
N(2)-Ga(1)-N(1)	176.23(4)
O(1)-Ga(1)-N(4)	169.12(4)
O(3)-Ga(1)-N(4)	89.13(3)
N(2)-Ga(1)-N(4)	96.00(4)
N(1)-Ga(1)-N(4)	81.24(4)
O(1)-Ga(1)-N(3)	90.75(3)
O(3)-Ga(1)-N(3)	168.39(3)
N(2)-Ga(1)-N(3)	81.60(4)
N(1)-Ga(1)-N(3)	95.49(4)
N(4)-Ga(1)-N(3)	83.17(4)
C(1)-O(1)-Ga(1)	127.88(6)
C(2)-O(2)-C(21)	116.02(8)
C(16)-O(3)-Ga(1)	125.78(6)
C(17)-O(4)-C(24)	113.63(8)
C(7)-N(1)-C(8)	119.53(9)

C(7)-N(1)-Ga(1)	124.61(7)
C(8)-N(1)-Ga(1)	115.39(7)
C(14)-N(2)-C(13)	119.89(9)
C(14)-N(2)-Ga(1)	123.58(7)
C(13)-N(2)-Ga(1)	115.69(7)
C(11)-N(3)-C(12)	115.98(10)
C(11)-N(3)-Ga(1)	109.54(7)
C(12)-N(3)-Ga(1)	108.46(7)
C(11)-N(3)-H(3)	106.9(10)
C(12)-N(3)-H(3)	107.8(10)
Ga(1)-N(3)-H(3)	107.9(10)
C(9)-N(4)-C(10)	116.33(10)
C(9)-N(4)-Ga(1)	108.07(7)
C(10)-N(4)-Ga(1)	109.92(7)
C(9)-N(4)-H(4)	108.1(10)
C(10)-N(4)-H(4)	107.3(10)
Ga(1)-N(4)-H(4)	106.7(10)
O(1)-C(1)-C(6)	124.60(9)
O(1)-C(1)-C(2)	118.17(9)
C(6)-C(1)-C(2)	117.20(9)
O(2)-C(2)-C(3)	119.03(9)
O(2)-C(2)-C(1)	119.41(9)
C(3)-C(2)-C(1)	121.27(9)
C(2)-C(3)-C(4)	120.71(10)
C(2)-C(3)-H(3A)	119.6
C(4)-C(3)-H(3A)	119.6
C(5)-C(4)-C(3)	119.67(10)
C(5)-C(4)-H(4A)	120.2
C(3)-C(4)-H(4A)	120.2
C(4)-C(5)-C(6)	120.76(10)
C(4)-C(5)-H(5)	119.6
C(6)-C(5)-H(5)	119.6
C(5)-C(6)-C(1)	120.33(9)
C(5)-C(6)-C(7)	116.45(9)
C(1)-C(6)-C(7)	123.17(9)
N(1)-C(7)-C(6)	125.33(9)

N(1)-C(7)-H(7)	117.3
C(6)-C(7)-H(7)	117.3
N(1)-C(8)-C(9)	107.87(9)
N(1)-C(8)-H(8A)	110.1
C(9)-C(8)-H(8A)	110.1
N(1)-C(8)-H(8B)	110.1
C(9)-C(8)-H(8B)	110.1
H(8A)-C(8)-H(8B)	108.4
N(4)-C(9)-C(8)	110.72(9)
N(4)-C(9)-H(9A)	109.5
C(8)-C(9)-H(9A)	109.5
N(4)-C(9)-H(9B)	109.5
C(8)-C(9)-H(9B)	109.5
H(9A)-C(9)-H(9B)	108.1
N(4)-C(10)-C(11)	111.53(9)
N(4)-C(10)-H(10A)	109.3
C(11)-C(10)-H(10A)	109.3
N(4)-C(10)-H(10B)	109.3
C(11)-C(10)-H(10B)	109.3
H(10A)-C(10)-H(10B)	108.0
N(3)-C(11)-C(10)	111.17(10)
N(3)-C(11)-H(11A)	109.4
C(10)-C(11)-H(11A)	109.4
N(3)-C(11)-H(11B)	109.4
C(10)-C(11)-H(11B)	109.4
H(11A)-C(11)-H(11B)	108.0
N(3)-C(12)-C(13)	110.93(9)
N(3)-C(12)-H(12A)	109.5
C(13)-C(12)-H(12A)	109.5
N(3)-C(12)-H(12B)	109.5
C(13)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	108.0
N(2)-C(13)-C(12)	107.63(9)
N(2)-C(13)-H(13A)	110.2
C(12)-C(13)-H(13A)	110.2
N(2)-C(13)-H(13B)	110.2

C(12)-C(13)-H(13B)	110.2
H(13A)-C(13)-H(13B)	108.5
N(2)-C(14)-C(15)	124.85(9)
N(2)-C(14)-H(14)	117.6
C(15)-C(14)-H(14)	117.6
C(20)-C(15)-C(16)	120.35(9)
C(20)-C(15)-C(14)	117.12(9)
C(16)-C(15)-C(14)	122.47(9)
O(3)-C(16)-C(15)	124.90(9)
O(3)-C(16)-C(17)	118.56(9)
C(15)-C(16)-C(17)	116.53(9)
C(18)-C(17)-O(4)	119.59(9)
C(18)-C(17)-C(16)	121.83(9)
O(4)-C(17)-C(16)	118.45(9)
C(17)-C(18)-C(19)	120.46(10)
C(17)-C(18)-H(18)	119.8
C(19)-C(18)-H(18)	119.8
C(20)-C(19)-C(18)	119.22(11)
C(20)-C(19)-H(19)	120.4
C(18)-C(19)-H(19)	120.4
C(19)-C(20)-C(15)	121.10(10)
C(19)-C(20)-H(20)	119.4
C(15)-C(20)-H(20)	119.4
O(2)-C(21)-C(23)	114.0(2)
O(2)-C(21)-C(22')	111.5(3)
O(2)-C(21)-C(22)	101.2(2)
C(23)-C(21)-C(22)	113.73(17)
O(2)-C(21)-C(23')	103.5(3)
C(22')-C(21)-C(23')	112.3(2)
O(2)-C(21)-H(21)	109.2
C(23)-C(21)-H(21)	109.2
C(22)-C(21)-H(21)	109.2
C(21)-C(22)-H(22A)	109.5
C(21)-C(22)-H(22B)	109.5
H(22A)-C(22)-H(22B)	109.5
C(21)-C(22)-H(22C)	109.5

H(22A)-C(22)-H(22C)	109.5
H(22B)-C(22)-H(22C)	109.5
C(21)-C(23)-H(23A)	109.5
C(21)-C(23)-H(23B)	109.5
H(23A)-C(23)-H(23B)	109.5
C(21)-C(23)-H(23C)	109.5
H(23A)-C(23)-H(23C)	109.5
H(23B)-C(23)-H(23C)	109.5
C(21)-C(22')-H(22D)	109.5
C(21)-C(22')-H(22E)	109.5
H(22D)-C(22')-H(22E)	109.5
C(21)-C(22')-H(22F)	109.5
H(22D)-C(22')-H(22F)	109.5
H(22E)-C(22')-H(22F)	109.5
C(21)-C(23')-H(23D)	109.5
C(21)-C(23')-H(23E)	109.5
H(23D)-C(23')-H(23E)	109.5
C(21)-C(23')-H(23F)	109.5
H(23D)-C(23')-H(23F)	109.5
H(23E)-C(23')-H(23F)	109.5
O(4)-C(24)-C(26)	106.02(9)
O(4)-C(24)-C(25)	109.34(9)
C(26)-C(24)-C(25)	113.84(10)
O(4)-C(24)-H(24)	109.2
C(26)-C(24)-H(24)	109.2
C(25)-C(24)-H(24)	109.2
C(24)-C(25)-H(25A)	109.5
C(24)-C(25)-H(25B)	109.5
H(25A)-C(25)-H(25B)	109.5
C(24)-C(25)-H(25C)	109.5
H(25A)-C(25)-H(25C)	109.5
H(25B)-C(25)-H(25C)	109.5
C(24)-C(26)-H(26A)	109.5
C(24)-C(26)-H(26B)	109.5
H(26A)-C(26)-H(26B)	109.5
C(24)-C(26)-H(26C)	109.5

H(26A)-C(26)-H(26C)	109.5
H(26B)-C(26)-H(26C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **5a**. 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}
I(1)	17(1)	18(1)	25(1)	0(1)	-1(1)	3(1)
I(1')	10(1)	11(1)	12(1)	1(1)	2(1)	1(1)
Ga(1)	12(1)	8(1)	10(1)	0(1)	5(1)	-2(1)
O(1)	14(1)	9(1)	13(1)	3(1)	3(1)	-2(1)
O(2)	14(1)	13(1)	17(1)	-1(1)	1(1)	-3(1)
O(3)	12(1)	16(1)	11(1)	-1(1)	4(1)	1(1)
O(4)	17(1)	12(1)	11(1)	2(1)	3(1)	-1(1)
N(1)	11(1)	13(1)	17(1)	-2(1)	6(1)	-2(1)
N(2)	19(1)	11(1)	10(1)	1(1)	4(1)	0(1)
N(3)	23(1)	10(1)	14(1)	-1(1)	9(1)	-3(1)
N(4)	19(1)	11(1)	17(1)	-3(1)	9(1)	-4(1)
C(1)	14(1)	10(1)	9(1)	0(1)	3(1)	-2(1)
C(2)	14(1)	11(1)	12(1)	0(1)	3(1)	-2(1)
C(3)	20(1)	11(1)	17(1)	2(1)	5(1)	-4(1)
C(4)	22(1)	12(1)	18(1)	4(1)	5(1)	0(1)
C(5)	18(1)	13(1)	14(1)	2(1)	4(1)	2(1)
C(6)	14(1)	11(1)	11(1)	1(1)	4(1)	0(1)
C(7)	13(1)	14(1)	14(1)	-1(1)	5(1)	1(1)
C(8)	13(1)	18(1)	32(1)	-2(1)	10(1)	-3(1)
C(9)	16(1)	17(1)	28(1)	-4(1)	7(1)	-6(1)
C(10)	40(1)	17(1)	22(1)	-4(1)	19(1)	-14(1)
C(11)	37(1)	20(1)	22(1)	-6(1)	21(1)	-12(1)
C(12)	32(1)	18(1)	11(1)	0(1)	5(1)	3(1)
C(13)	32(1)	16(1)	12(1)	3(1)	6(1)	5(1)
C(14)	16(1)	13(1)	13(1)	-1(1)	1(1)	1(1)
C(15)	14(1)	13(1)	13(1)	-1(1)	3(1)	1(1)
C(16)	13(1)	9(1)	11(1)	-1(1)	4(1)	0(1)
C(17)	16(1)	11(1)	12(1)	0(1)	5(1)	1(1)
C(18)	22(1)	20(1)	17(1)	0(1)	11(1)	4(1)
C(19)	19(1)	28(1)	24(1)	-1(1)	12(1)	6(1)
C(20)	15(1)	21(1)	20(1)	-1(1)	5(1)	5(1)

C(21)	15(1)	15(1)	29(1)	0(1)	6(1)	-1(1)
C(22)	18(1)	24(1)	38(1)	8(1)	13(1)	11(1)
C(23)	18(1)	24(1)	38(1)	8(1)	13(1)	11(1)
C(22')	18(1)	24(1)	38(1)	8(1)	13(1)	11(1)
C(23')	18(1)	24(1)	38(1)	8(1)	13(1)	11(1)
C(24)	20(1)	13(1)	11(1)	0(1)	3(1)	-4(1)
C(25)	34(1)	15(1)	19(1)	-5(1)	6(1)	-4(1)
C(26)	19(1)	24(1)	25(1)	4(1)	4(1)	-1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for **5a**.

	x	y	z	U(eq)
H(3A)	4397	9977	4009	19
H(4A)	2325	10338	3165	22
H(5)	685	9496	3008	18
H(7)	-124	8353	3305	16
H(8A)	-1118	7240	3245	25
H(8B)	-805	7151	4369	25
H(9A)	-915	5918	3831	24
H(9B)	-145	6114	3129	24
H(10A)	1494	5426	5757	29
H(10B)	31	5635	5379	29
H(11A)	399	6860	5903	28
H(11B)	1311	6372	6755	28
H(12A)	3395	6577	7296	25
H(12B)	4069	7164	6801	25
H(13A)	4950	6021	6635	25
H(13B)	3623	5590	6355	25
H(14)	5452	5853	5326	18
H(18)	4418	6220	1392	22
H(19)	6356	6003	2586	27
H(20)	6425	5906	4155	22
H(21)	5122	7757	4300	24
H(22A)	7166	8180	5950	39
H(22B)	7018	7355	5485	39
H(22C)	6012	7653	5950	39
H(23A)	5996	8614	3455	39
H(23B)	6881	7893	3821	39
H(23C)	7226	8683	4368	39
H(22D)	7370	8114	5815	39
H(22E)	7128	7331	5247	39
H(22F)	6305	7549	5902	39

H(23D)	5762	8783	3476	39
H(23E)	6740	8097	3665	39
H(23F)	7070	8834	4323	39
H(24)	1332	5744	2121	18
H(25A)	1579	5392	319	35
H(25B)	936	4845	881	35
H(25C)	2407	5023	1304	35
H(26A)	-42	6720	1347	35
H(26B)	-618	5921	911	35
H(26C)	44	6453	346	35
H(3)	2125(14)	7414(9)	6114(11)	18(4)
H(4)	1259(15)	5595(8)	4311(11)	18(4)

Table 6. Torsion angles [°] for **5a.**

Ga(1)-O(1)-C(1)-C(6)	18.17(14)
Ga(1)-O(1)-C(1)-C(2)	-163.70(7)
C(21)-O(2)-C(2)-C(3)	-119.45(11)
C(21)-O(2)-C(2)-C(1)	66.59(12)
O(1)-C(1)-C(2)-O(2)	-2.40(14)
C(6)-C(1)-C(2)-O(2)	175.87(9)
O(1)-C(1)-C(2)-C(3)	-176.21(9)
C(6)-C(1)-C(2)-C(3)	2.06(15)
O(2)-C(2)-C(3)-C(4)	-174.23(10)
C(1)-C(2)-C(3)-C(4)	-0.39(16)
C(2)-C(3)-C(4)-C(5)	-0.95(17)
C(3)-C(4)-C(5)-C(6)	0.54(17)
C(4)-C(5)-C(6)-C(1)	1.20(16)
C(4)-C(5)-C(6)-C(7)	178.83(10)
O(1)-C(1)-C(6)-C(5)	175.70(9)
C(2)-C(1)-C(6)-C(5)	-2.44(14)
O(1)-C(1)-C(6)-C(7)	-1.76(16)
C(2)-C(1)-C(6)-C(7)	-179.90(9)
C(8)-N(1)-C(7)-C(6)	178.83(10)
Ga(1)-N(1)-C(7)-C(6)	-9.38(15)
C(5)-C(6)-C(7)-N(1)	-179.63(10)
C(1)-C(6)-C(7)-N(1)	-2.08(16)
C(7)-N(1)-C(8)-C(9)	147.50(10)
Ga(1)-N(1)-C(8)-C(9)	-25.03(12)
C(10)-N(4)-C(9)-C(8)	82.81(12)
Ga(1)-N(4)-C(9)-C(8)	-41.34(11)
N(1)-C(8)-C(9)-N(4)	43.57(13)
C(9)-N(4)-C(10)-C(11)	-92.21(13)
Ga(1)-N(4)-C(10)-C(11)	30.98(13)
C(12)-N(3)-C(11)-C(10)	-89.45(12)
Ga(1)-N(3)-C(11)-C(10)	33.68(13)
N(4)-C(10)-C(11)-N(3)	-43.63(16)
C(11)-N(3)-C(12)-C(13)	86.03(12)
Ga(1)-N(3)-C(12)-C(13)	-37.66(11)

C(14)-N(2)-C(13)-C(12)	141.13(11)
Ga(1)-N(2)-C(13)-C(12)	-28.74(12)
N(3)-C(12)-C(13)-N(2)	43.27(13)
C(13)-N(2)-C(14)-C(15)	178.00(10)
Ga(1)-N(2)-C(14)-C(15)	-12.97(15)
N(2)-C(14)-C(15)-C(20)	173.45(10)
N(2)-C(14)-C(15)-C(16)	-9.30(16)
Ga(1)-O(3)-C(16)-C(15)	15.72(14)
Ga(1)-O(3)-C(16)-C(17)	-165.44(7)
C(20)-C(15)-C(16)-O(3)	-174.19(10)
C(14)-C(15)-C(16)-O(3)	8.65(16)
C(20)-C(15)-C(16)-C(17)	6.95(14)
C(14)-C(15)-C(16)-C(17)	-170.21(9)
C(24)-O(4)-C(17)-C(18)	115.69(11)
C(24)-O(4)-C(17)-C(16)	-68.41(11)
O(3)-C(16)-C(17)-C(18)	173.12(10)
C(15)-C(16)-C(17)-C(18)	-7.94(15)
O(3)-C(16)-C(17)-O(4)	-2.68(14)
C(15)-C(16)-C(17)-O(4)	176.26(9)
O(4)-C(17)-C(18)-C(19)	179.07(10)
C(16)-C(17)-C(18)-C(19)	3.32(17)
C(17)-C(18)-C(19)-C(20)	2.57(18)
C(18)-C(19)-C(20)-C(15)	-3.47(19)
C(16)-C(15)-C(20)-C(19)	-1.45(17)
C(14)-C(15)-C(20)-C(19)	175.86(11)
C(2)-O(2)-C(21)-C(23)	92.4(2)
C(2)-O(2)-C(21)-C(22')	-150.8(3)
C(2)-O(2)-C(21)-C(22)	-145.1(2)
C(2)-O(2)-C(21)-C(23')	88.3(2)
C(17)-O(4)-C(24)-C(26)	158.64(9)
C(17)-O(4)-C(24)-C(25)	-78.24(10)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for **5a** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(7)-H(7)...I(1)#1	0.95	3.31	4.1529(14)	148.8
C(10)-H(10B)...I(1)#2	0.99	3.04	3.8444(17)	138.8
C(14)-H(14)...I(1)#3	0.95	2.93	3.8427(15)	160.5
C(18)-H(18)...O(2)#4	0.95	2.58	3.4805(14)	158.7
C(21)-H(21)...O(1)	1.00	2.40	2.9550(14)	114.2
C(22)-H(22B)...I(1)#3	0.98	3.31	4.125(5)	141.9
C(24)-H(24)...I(1)	1.00	3.27	4.122(2)	143.6
C(24)-H(24)...O(3)	1.00	2.40	2.9815(12)	116.5
N(3)-H(3)...O(4)#5	0.846(15)	2.047(15)	2.8764(12)	166.5(14)
N(4)-H(4)...I(1)	0.871(15)	2.725(16)	3.5910(14)	173.3(14)

Symmetry transformations used to generate equivalent atoms:

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

#4 x,-y+3/2,z-1/2 #5 x,-y+3/2,z+1/2

Figure Legends

Figure 1. Projection view with 50% probability ellipsoids for **5a** (disorder components omitted for clarity).