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Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **11**.

	x	y	z	U(eq)
H(01)	7633(1)	10378(6)	4162(1)	56
H(08)	7891(2)	10289(10)	6566(2)	75
H(10)	5861(2)	10533(8)	4036(1)	68
H(12)	6325(2)	12026(7)	6114(1)	54
H(13)	5760(2)	9422(7)	5418(1)	52
H(14)	6169(2)	7025(7)	5996(2)	57
H(15)	5679(2)	8903(6)	6802(1)	52
H(16A)	6112(2)	5757(8)	6995(2)	68
H(16B)	6871(2)	6392(8)	6911(2)	68
H(17A)	7095(3)	8170(7)	7649(2)	74
H(17B)	6282(3)	8035(7)	7697(2)	74
H(21A)	7202(5)	995(10)	9289(3)	164
H(21B)	6959(5)	3022(10)	9420(3)	164
H(21C)	7766(5)	2656(10)	9387(3)	164
H(22A)	7531(4)	2259(9)	8020(3)	147
H(22B)	7664(4)	612(9)	8437(3)	147
H(22C)	8115(4)	2485(9)	8558(3)	147
H(23A)	6228(4)	2102(9)	8013(3)	145
H(23B)	5957(4)	2443(9)	8535(3)	145
H(23C)	6353(4)	554(9)	8459(3)	145
H(25)	6811(4)	7156(13)	9560(3)	118
H(26)	5850(5)	8320(17)	9855(4)	147
H(27)	4727(5)	8497(13)	9303(4)	133
H(28)	4546(3)	7413(13)	8454(3)	122
H(29)	5493(3)	6114(11)	8152(2)	94
H(31)	8232(3)	6655(10)	8146(2)	84
H(32)	9385(3)	7859(13)	8465(3)	111
H(33)	9788(3)	8596(12)	9340(3)	114
H(34)	9055(4)	7989(16)	9915(3)	136
H(35)	7925(3)	6671(13)	9614(2)	106
H(36A)	4846(4)	6832(12)	5616(3)	109
H(36B)	5232(4)	5307(12)	6025(3)	109
H(37)	4989(1)	11351(6)	5570(1)	96
H(39)	5326(3)	10753(13)	3213(2)	61(13)
H(40A)	5670(4)	13773(17)	3086(3)	164
H(40B)	5370(4)	12934(17)	2515(3)	164
H(40C)	6212(4)	13096(17)	2748(3)	164
H(41A)	5719(5)	8177(17)	2804(2)	194
H(41B)	6192(5)	9478(17)	2527(2)	194
H(41C)	5340(5)	9592(17)	2358(2)	194
H(43)	4909(2)	7394(8)	6587(2)	102
H(43A)	9900(7)	298(23)	6462(6)	99

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compound **11** cif file

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J4531-4/4

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are based on F, with F set to zero for negative F^2^. R-factors based
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factors based on ALL data will be even larger.
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O11 O 0.65821(14) 0.9924(5) 0.66275(9) 0.0559(8) Uani 1 d . .
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H13 H 0.5760(2) 0.9422(7) 0.54177(13) 0.052 Uiso 1 calc R .
C14 C 0.5791(2) 0.7792(7) 0.60965(15) 0.0475(8) Uani 1 d . .
H14 H 0.6169(2) 0.7025(7) 0.59955(15) 0.057 Uiso 1 calc R .
C15 C 0.6081(2) 0.8396(6) 0.66638(14) 0.0430(8) Uani 1 d . .
H15 H 0.5679(2) 0.8903(6) 0.68021(14) 0.052 Uiso 1 calc R .
C16 C 0.6446(2) 0.6814(8) 0.7029(2) 0.0568(10) Uani 1 d . .
H16A H 0.6112(2) 0.5757(8) 0.6995(2) 0.068 Uiso 1 calc R .
H16B H 0.6871(2) 0.6392(8) 0.6911(2) 0.068 Uiso 1 calc R .
C17 C 0.6672(3) 0.7354(7) 0.7593(2) 0.0621(11) Uani 1 d . .
H17A H 0.7095(3) 0.8170(7) 0.7649(2) 0.074 Uiso 1 calc R .
H17B H 0.6282(3) 0.8035(7) 0.7697(2) 0.074 Uiso 1 calc R .
O18 O 0.6841(2) 0.5681(5) 0.79037(10) 0.0597(8) Uani 1 d . .
Si19 Si 0.70261(6) 0.5419(2) 0.85506(4) 0.0459(3) Uani 1 d . .
C20 C 0.7067(3) 0.2787(7) 0.8653(2) 0.0640(11) Uani 1 d . .
C21 C 0.7267(5) 0.2323(10) 0.9241(3) 0.109(3) Uani 1 d . .
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C22 C 0.7648(4) 0.1959(9) 0.8393(3) 0.098(2) Uani 1 d . .
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H23C H 0.6353(4) 0.0554(9) 0.8459(3) 0.145 Uiso 1 calc R .
C24 C 0.6273(3) 0.6486(6) 0.8817(2) 0.0557(10) Uani 1 d . .
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H25 H 0.6811(4) 0.7156(13) 0.9560(3) 0.118 Uiso 1 calc R .
C26 C 0.5778(5) 0.7895(17) 0.9506(4) 0.123(3) Uani 1 d . .
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C27 C 0.5113(5) 0.7988(13) 0.9182(4) 0.110(3) Uani 1 d . .
H27 H 0.4727(5) 0.8497(13) 0.9303(4) 0.133 Uiso 1 calc R .
C28 C 0.5007(3) 0.7345(13) 0.8681(3) 0.102(3) Uani 1 d . .
H28 H 0.4546(3) 0.7413(13) 0.8454(3) 0.122 Uiso 1 calc R .
C29 C 0.5580(3) 0.6577(11) 0.8498(2) 0.078(2) Uani 1 d . .
H29 H 0.5493(3) 0.6114(11) 0.8152(2) 0.094 Uiso 1 calc R .
C30 C 0.7937(2) 0.6497(6) 0.8842(2) 0.0527(9) Uani 1 d . .
C31 C 0.8393(3) 0.6895(10) 0.8508(2) 0.0704(13) Uani 1 d . .
H31 H 0.8232(3) 0.6655(10) 0.8146(2) 0.084 Uiso 1 calc R .
C32 C 0.9087(3) 0.7647(13) 0.8700(3) 0.093(2) Uani 1 d . .
H32 H 0.9385(3) 0.7859(13) 0.8465(3) 0.111 Uiso 1 calc R .
C33 C 0.9330(3) 0.8067(12) 0.9215(3) 0.095(2) Uani 1 d . .
H33 H 0.9788(3) 0.8596(12) 0.9340(3) 0.114 Uiso 1 calc R .

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C34 C 0.8892(4) 0.7705(16) 0.9555(3) 0.113(3) Uani 1 d . .
H34 H 0.9055(4) 0.7989(16) 0.9915(3) 0.136 Uiso 1 calc R .
C35 C 0.8209(3) 0.6920(13) 0.9372(2) 0.089(2) Uani 1 d . .
H35 H 0.7925(3) 0.6671(13) 0.9614(2) 0.106 Uiso 1 calc R .
C36 C 0.5104(4) 0.6634(12) 0.5983(3) 0.091(2) Uani 1 d . .
H36A H 0.4846(4) 0.6832(12) 0.5616(3) 0.109 Uiso 1 calc R 1
H36B H 0.5232(4) 0.5307(12) 0.6025(3) 0.109 Uiso 1 calc R 1
O37 O 0.51263(13) 1.0701(6) 0.58376(12) 0.0638(9) Uani 1 d . .
H37 H 0.49890(13) 1.1351(6) 0.55702(12) 0.096 Uiso 1 calc R .
C38 C 0.6429(2) 1.0677(11) 0.3516(2) 0.0687(15) Uani 1 d . .
C39 C 0.5759(3) 1.0903(13) 0.3069(2) 0.088(2) Uani 1 d . .
H39 H 0.5326(3) 1.0753(13) 0.3213(2) 0.061(13) Uiso 1 calc R .
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H43 H 0.4909(2) 0.7394(8) 0.6587(2) 0.102 Uiso 0.760(11) calc PR 1
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O1W O 1.0000 0.8110(6) 0.5000 0.0475(8) Uani 1 d S 1
O43 O 0.9899(7) 0.0180(23) 0.6146(6) 0.066(5) Uani 0.240(11) d P 2
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C21 0.181(8) 0.057(3) 0.082(4) 0.024(3) 0.014(4) -0.001(4)
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C25 0.091(4) 0.125(6) 0.081(4) -0.036(4) 0.025(3) 0.006(4)
C26 0.123(6) 0.144(8) 0.119(6) -0.047(6) 0.064(5) 0.002(6)
C27 0.101(5) 0.095(5) 0.158(7) 0.019(5) 0.077(5) 0.023(4)
C28 0.068(3) 0.117(6) 0.129(6) 0.048(5) 0.040(4) 0.022(4)
C29 0.065(3) 0.090(4) 0.081(3) 0.021(3) 0.019(2) 0.007(3)
C30 0.062(2) 0.045(2) 0.051(2) 0.004(2) 0.011(2) 0.002(2)
C31 0.065(3) 0.083(4) 0.065(3) 0.001(3) 0.019(2) 0.003(3)
C32 0.062(3) 0.116(6) 0.100(4) 0.015(4) 0.017(3) -0.006(4)
C33 0.062(3) 0.092(5) 0.119(5) 0.002(4) -0.001(3) -0.013(3)
C34 0.094(4) 0.148(8) 0.085(4) -0.029(5) -0.005(3) -0.029(5)
C35 0.082(3) 0.124(6) 0.058(3) -0.015(3) 0.012(2) -0.026(4)
C36 0.104(4) 0.101(5) 0.077(3) -0.025(3) 0.041(3) -0.056(4)
O37 0.0325(11) 0.095(3) 0.067(2) 0.031(2) 0.0185(11) 0.016(2)
C38 0.052(2) 0.111(5) 0.049(2) -0.003(3) 0.025(2) -0.023(3)
C39 0.067(3) 0.158(8) 0.041(2) 0.001(3) 0.017(2) -0.042(4)
C40 0.090(4) 0.154(8) 0.072(4) -0.007(5) -0.003(3) -0.014(5)
C41 0.161(7) 0.155(9) 0.062(3) -0.017(5) 0.007(4) -0.069(7)
O42 0.055(2) 0.178(5) 0.061(2) -0.011(3) 0.0327(13) -0.027(3)
O43 0.044(2) 0.084(4) 0.079(3) -0.010(3) 0.023(2) -0.012(2)
O44 0.0317(11) 0.091(3) 0.091(2) 0.034(2) 0.0297(12) 0.013(2)
O1W 0.039(2) 0.055(2) 0.049(2) 0.000 0.0130(14) 0.000
O43 0.064(8) 0.061(10) 0.071(8) 0.009(7) 0.013(6) -0.004(7)

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;
All esds (except the esd in the dihedral angle between two l.s. planes)
are estimated using the full covariance matrix. The cell esds are taken
into account individually in the estimation of esds in distances, angles
and torsion angles; correlations between esds in cell parameters are only
used when they are defined by crystal symmetry. An approximate (isotropic)
treatment of cell esds is used for estimating esds involving l.s. planes.
;

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N09 C12 C13 112.9(3) . . ?
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O37 C13 C12 107.4(4) . . ?
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C15 C14 C13 103.2(3) . . ?
C36 C14 C13 115.1(4) . . ?
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O18 C17 C16 108.5(4) . . ?
C17 O18 Si19 129.5(3) . . ?
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C29 C24 Si19 119.8(4) . . ?
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C27 C28 C29 120.5(7) . . ?
C24 C29 C28 121.5(6) . . ?
C35 C30 C31 115.6(5) . . ?
C35 C30 Si19 125.6(4) . . ?
C31 C30 Si19 118.8(4) . . ?
C30 C31 C32 122.0(5) . . ?
C33 C32 C31 120.9(6) . . ?
C32 C33 C34 118.7(6) . . ?
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O43 C36 C14 136.7(10) 3_455 . ?
O43 C36 C14 113.3(5) . . ?

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O42 C38 N10 122.1(4) . . ?
O42 C38 C39 122.1(4) . . ?
N10 C38 C39 115.8(3) . . ?
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J4531-51

Legends to Figures in Supplementary Material

Figure 1. Stereoscopic view of the crystal structure of bishomoguanosine derivative **11** (ORTEP). The major conformation (75%) of the disordered C-3"-hydroxyl group is depicted. Ellipsoids of the non-hydrogen atoms are shown at the 50% level.

Figure 2. Increase in UV absorption of partially protected oligomers dissolved in dichloromethane with increasing methanol content. Oligomers were dissolved in CH_2Cl_2/MeOH (2:1), which was separately shown to dissolve them fully by ^1H NMR, diluted with CH_2Cl_2 to 0.5% MeOH for the first measurement, and further methanol was added to the desired content. a) Selected spectra of octamer **55**; b) Plot of hyperchromicity at 260 nm against methanol content for octamer **55**, and tetramers **47** and **49**.

Figure 3. Mass spectra of fully deprotected oligomers. a) and b) MALDI-TOF spectra (2-(4-hydroxyphenylazo)benzoic acid matrix, 20 kV, linear, positive mode, insulin internal reference) of $ASO_2USO_2GSO_2G$ tetramer **57** (a), and $ASO_2USO_2GSO_2GSO_2USO_2CSO_2ASO_2U$ octamer **58** (b); parent peaks are Na^+-adducts; double Na^+-addition gives rise to higher mass peaks. c) Parent peak region of electrospray ionization spectrum of **58** (direct injection of H_2O/CH_3CN solution, elution with CH_3CN/TFA 0.1% aq/methoxyethanol/*i*-propanol) after transformation, yielding the average unprotonated mass.

Figure 4. UV absorbance *versus* temperature profile for **58**. Concentration of single-stranded oligosulfone not determined; the approximate concentration, based on an extinction coefficient calculated from the extinction coefficients of the constituent nucleobases in natural oligonucleotides is 2-3 μM. Spectrum obtained in 150 mM NaCl / 10 mM Tris-HCl pH 7. The third consecutive heating and cooling cycle (ca. 2 °C/min rate) is depicted.

Figure 5. UV absorbance *versus* temperature profile for 5'-rAUGGUCAU (Cruachem, Glasgow). 1.4 μM concentration of single-stranded oligonucleotide in 150 mM NaCl / 10 mM

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Tris-HCl pH 7. The second consecutive heating and cooling cycle (ca. 2 °C/min rate) is depicted.

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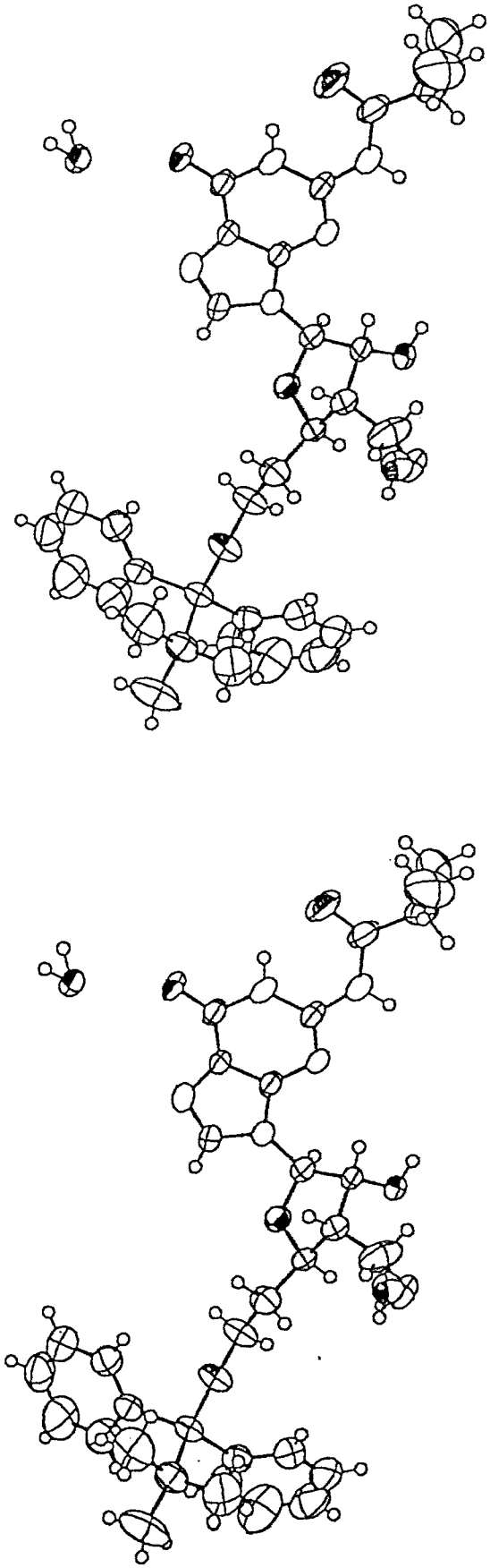

Figure 1

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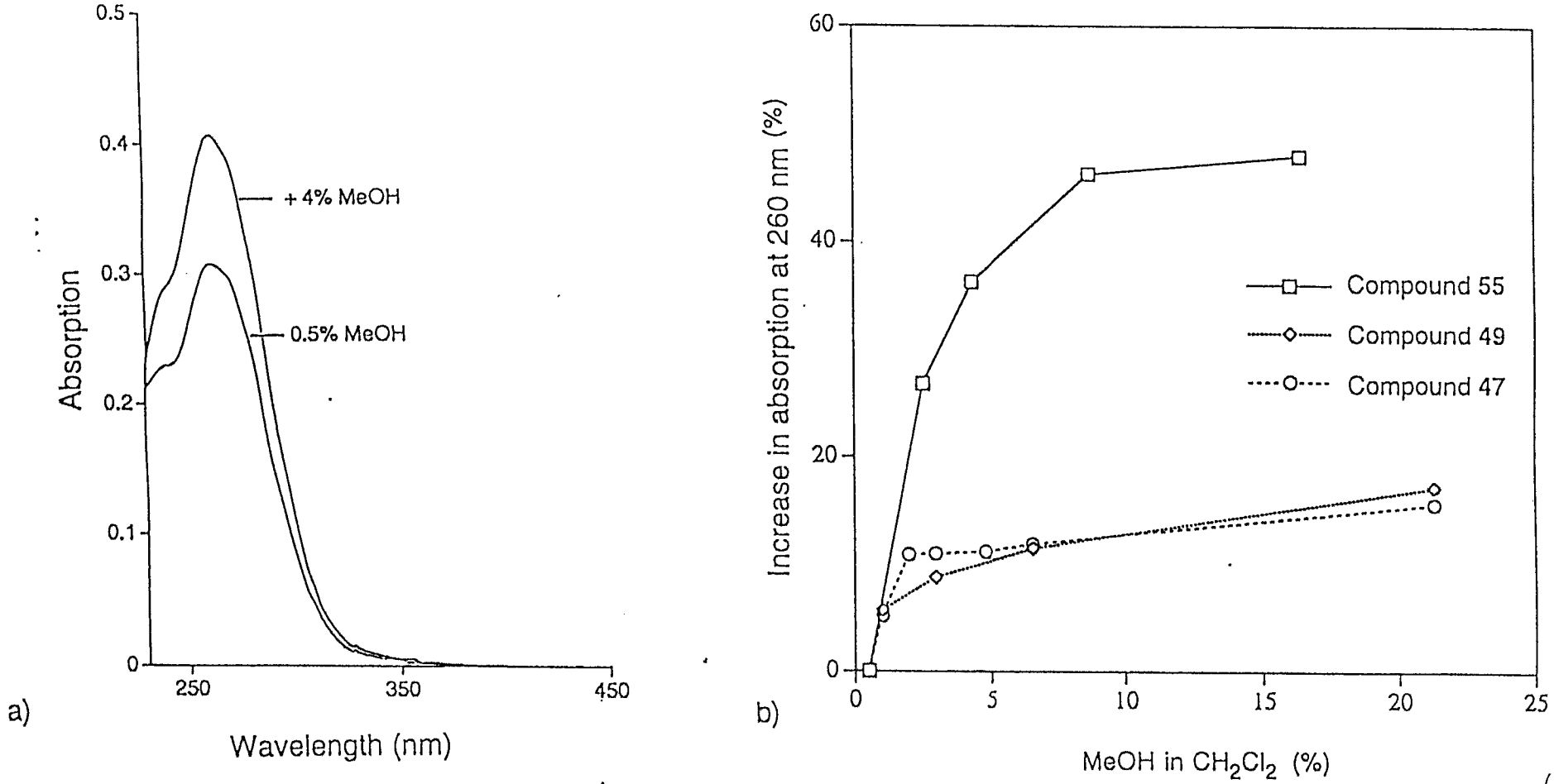

J4531-54

Figure 2

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J4531-55

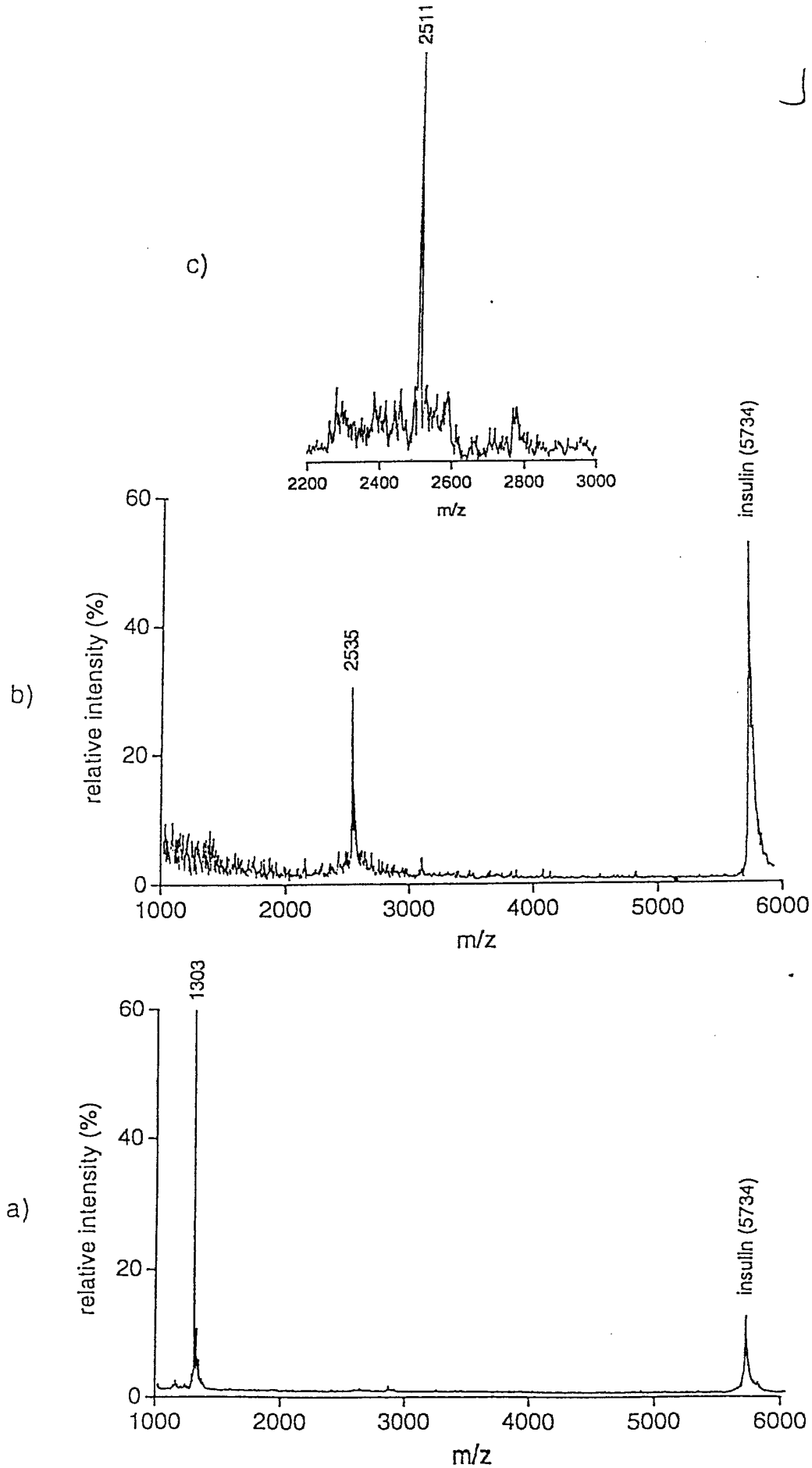

Figure 3

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J4531-56

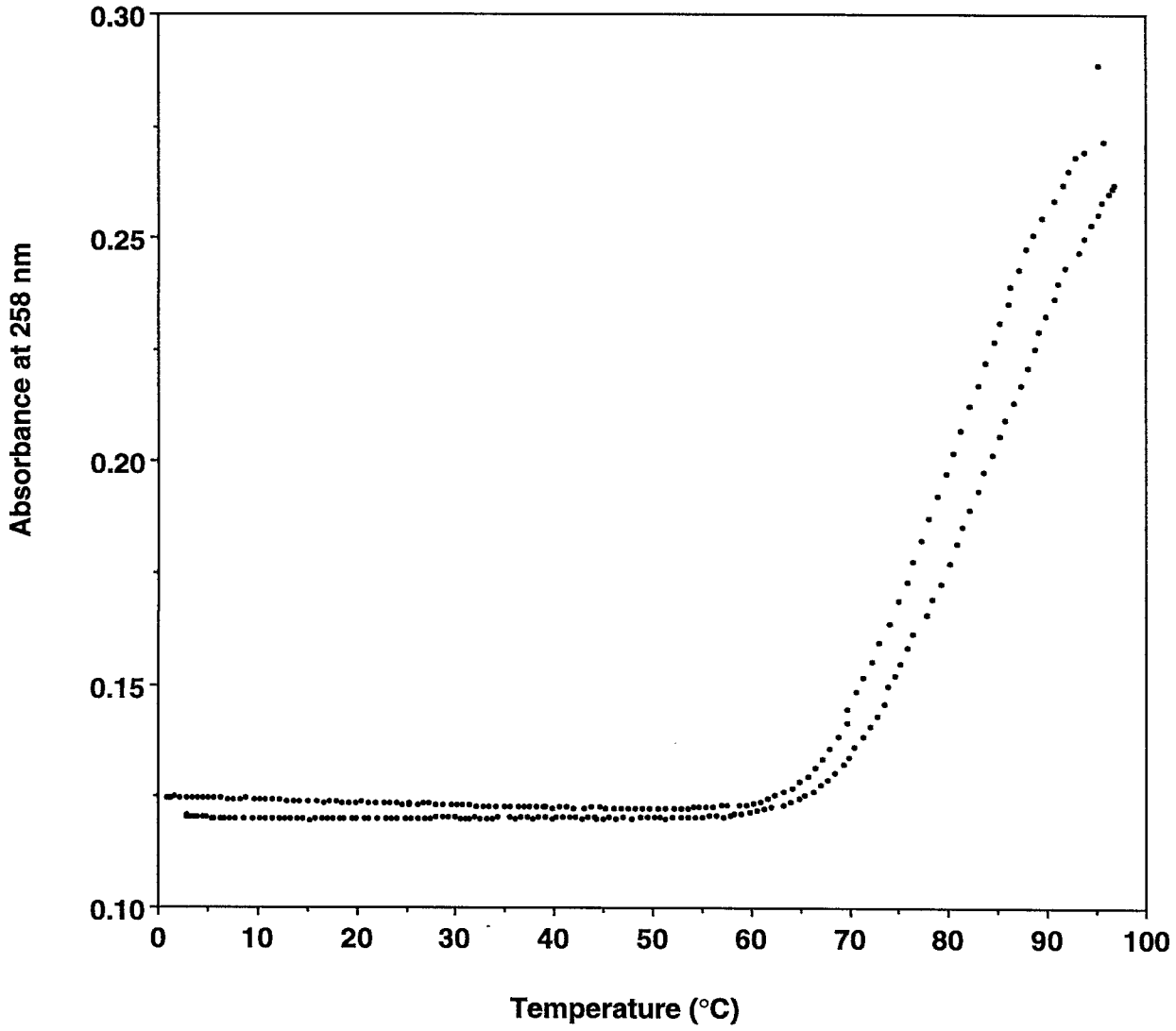

Figure 4

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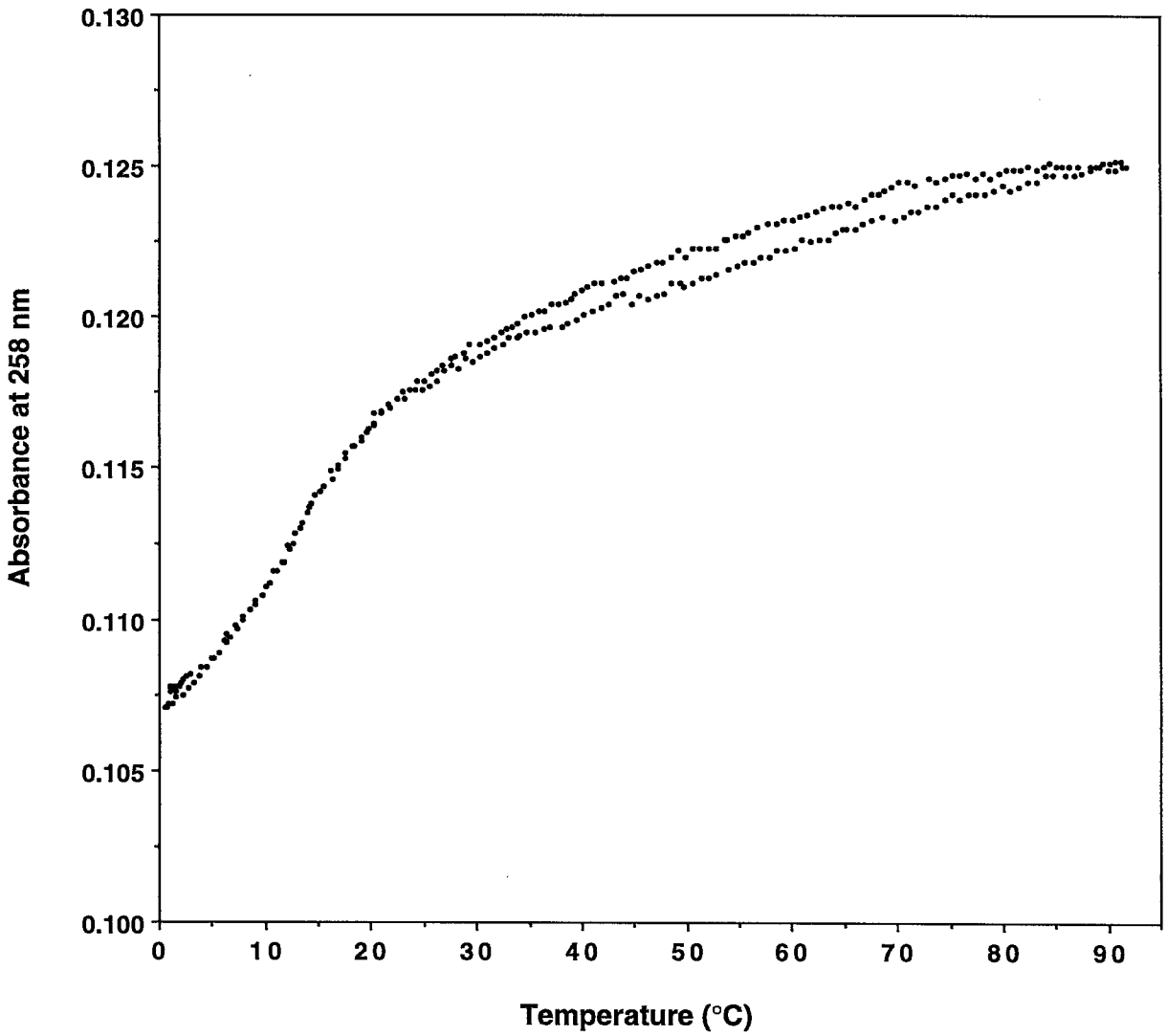

Figure 5

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