Characterization of a Novel Analog of 1α,25(OH)$_2$-Vitamin D$_3$ with Two Side-Chains: Interaction with its Nuclear Receptor and Cellular Actions

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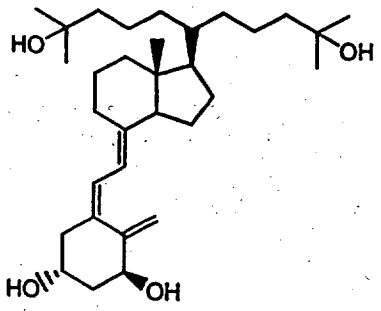
Supporting Information: (page 1 of 3 pages)

MATERIAL AND METHODS

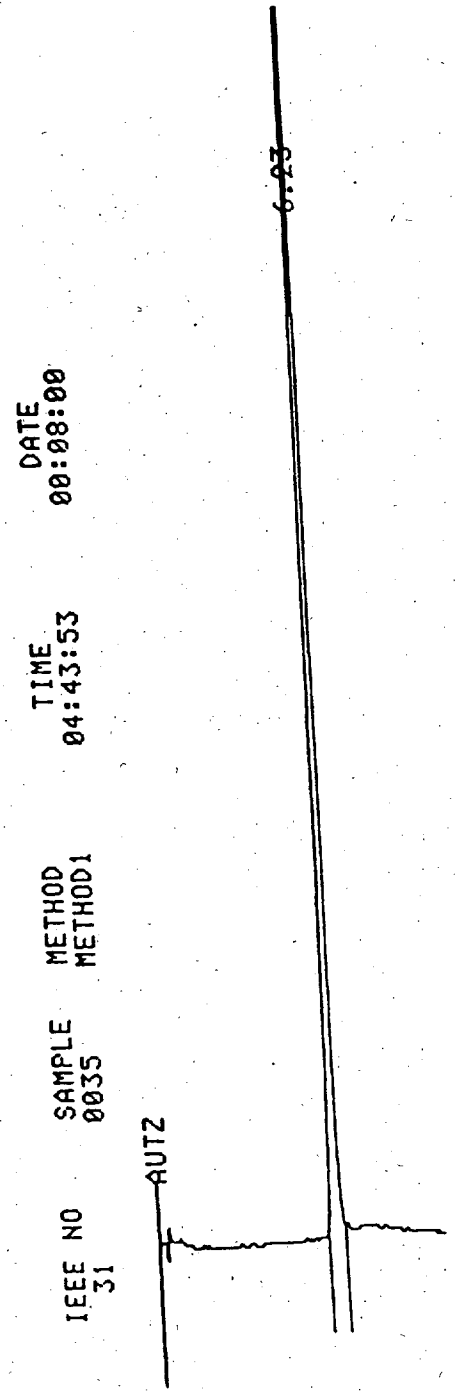
The NMR spectra were acquired using a Varian UNITYplus® 400 MHz spectrometer. The samples were dissolved in Deuterochloroform and the chemical shifts are referenced to Tetramethylsilane as internal zero standard.

Mass spectra data were measured using a VG AutoSpec, VG 7070E or Micromass Platform II mass spectrometer utilizing one of the following ionization methods: electron impact, fast atom bombardment, liquid secondary ion mass spectrometry, electrospray or atmospheric pressure chemical ionization. Accurate mass measurements were obtained at a minimum of 10,000 resolution.

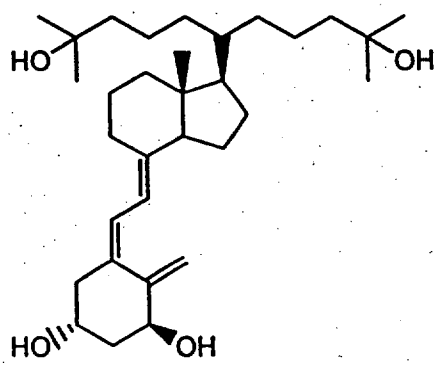
1,25-Dihydroxy-21-(3-hydroxy-3-methylbutyl)-cholecalciferol
(Ro 27-2310, Mw 502.8)



HPLC:
Column: Chromasil C-18
Size: 0.46 x 5 cm
Solvent System: H_20 - CH_3CN 20-65%
2 ml/min
Detector: UV diode array 215 nm



C-13 NMR Spectrum of 1,25-Dihydroxy-21-(3-hydroxy-3-methylbutyl)cholecalciferol (Gemini) in $CDCl_3$



δ ppm	
147.630	S
143.047	S
132.964	S
124.912	D
117.034	D
111.734	T
71.062	S
70.728	D
66.797	D
56.283	D
52.919	D
45.923	S
45.202	T
44.445	T
44.343	T
42.799	T
40.134	T
39.392	D
31.266	T
29.315	Q
29.213	Q
29.170	Q
29.053	T
27.044	T
23.578	T
22.107	T
20.083	T
19.792	T
12.089	Q

Number of protonated carbons: 24
CH: 7
CH2 13
CH3: 4
Number of quaternary carbons: 6. Two have the same chemical shift 71.062 ppm

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