

Identity and Purity:

Source of Material

The material was made by an unambiguous synthetic route, using literature procedures where possible; starting materials were purchased from reputable sources and all intermediates were checked for identity by NMR.

Proton NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available. Isotopic labelling: signals at the sites of deuteration are greatly diminished, compared the spectrum of unlabelled material.

Residual solvents: small amounts of dioxane (0.4 % w/w) and 2-ethoxyethanol (0.8 % w/w) are observed.

Impurities: no significant impurities are evident in the spectrum.

Carbon-13 NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available. Isotopic labelling: signals at the sites of deuteration have greatly diminished compared with the spectrum of unlabelled material, indicating clean deuteration.

Residual solvents: signals for dioxane and 2-ethoxyethanol are observed.

High-resolution mass spectrum (ESI+): found m/z 257.1355, $z = 2$. $C_{22}H_{24}D_8Cl_2N_{10}$ $[M+2H]^{2+}$ requires m/z 257.1341, $z = 2$. The deviation of 5.3 ppm is slightly outside normally accepted limits for the establishment of identity by HRMS, and the mass spectral data should be considered in conjunction with other identity criteria. No signal for d_0 material was seen (detection limit about 0.5 %).

HPLC: A sharp, slightly tailing peak is observed (95.6 area %). Note: in the absence of reference materials for preparing calibration curves, it is assumed that all peaks have the same detector response. Where possible, the conditions of analysis follow a pharmacopeial or literature method, or have been adapted from same.

Elemental Analysis: Found: C 45.21, H 4.20, D 2.86, N 23.93 %

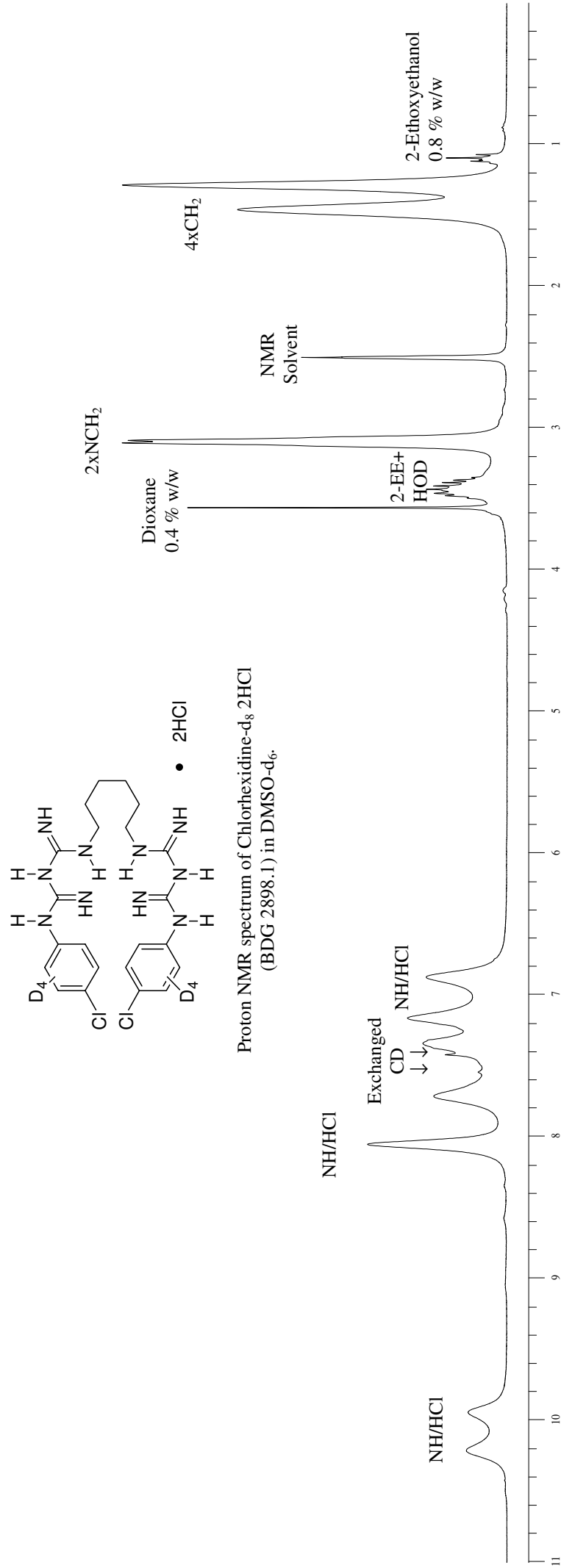
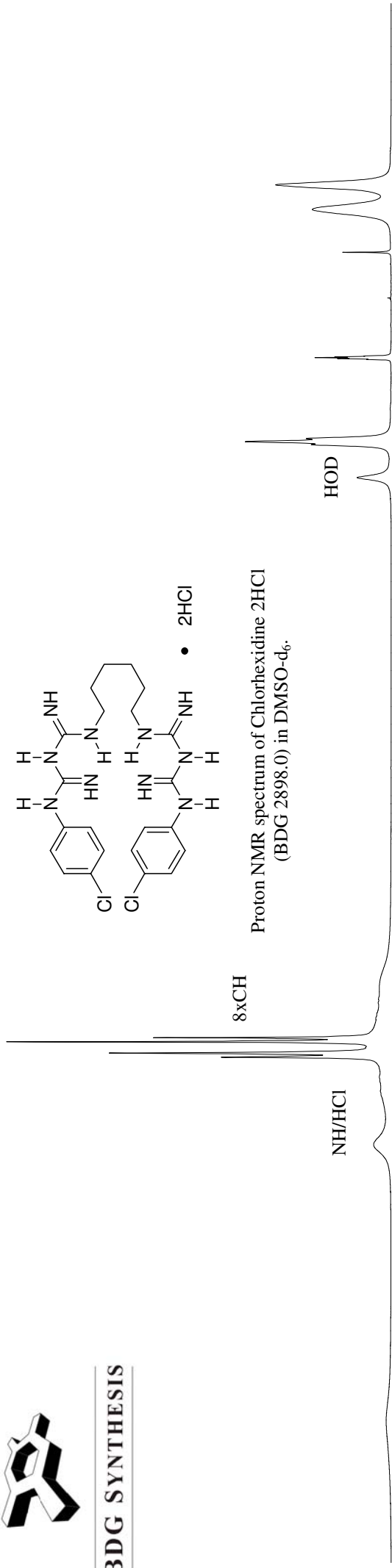
$C_{22}H_{22}D_8Cl_2N_{10} \cdot 2HCl$ requires: C 45.06, H 4.13, D 2.75, N 23.89 %

The elemental analyses fall within generally accepted limits for establishing the molecular formula given.

The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

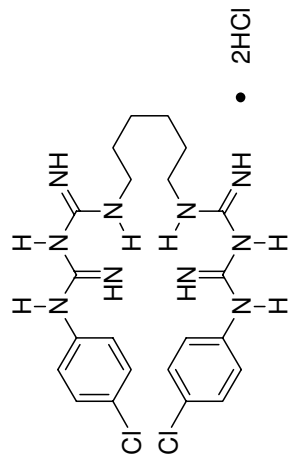


BDG SYNTHESIS

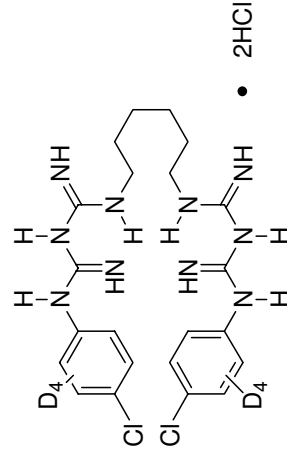
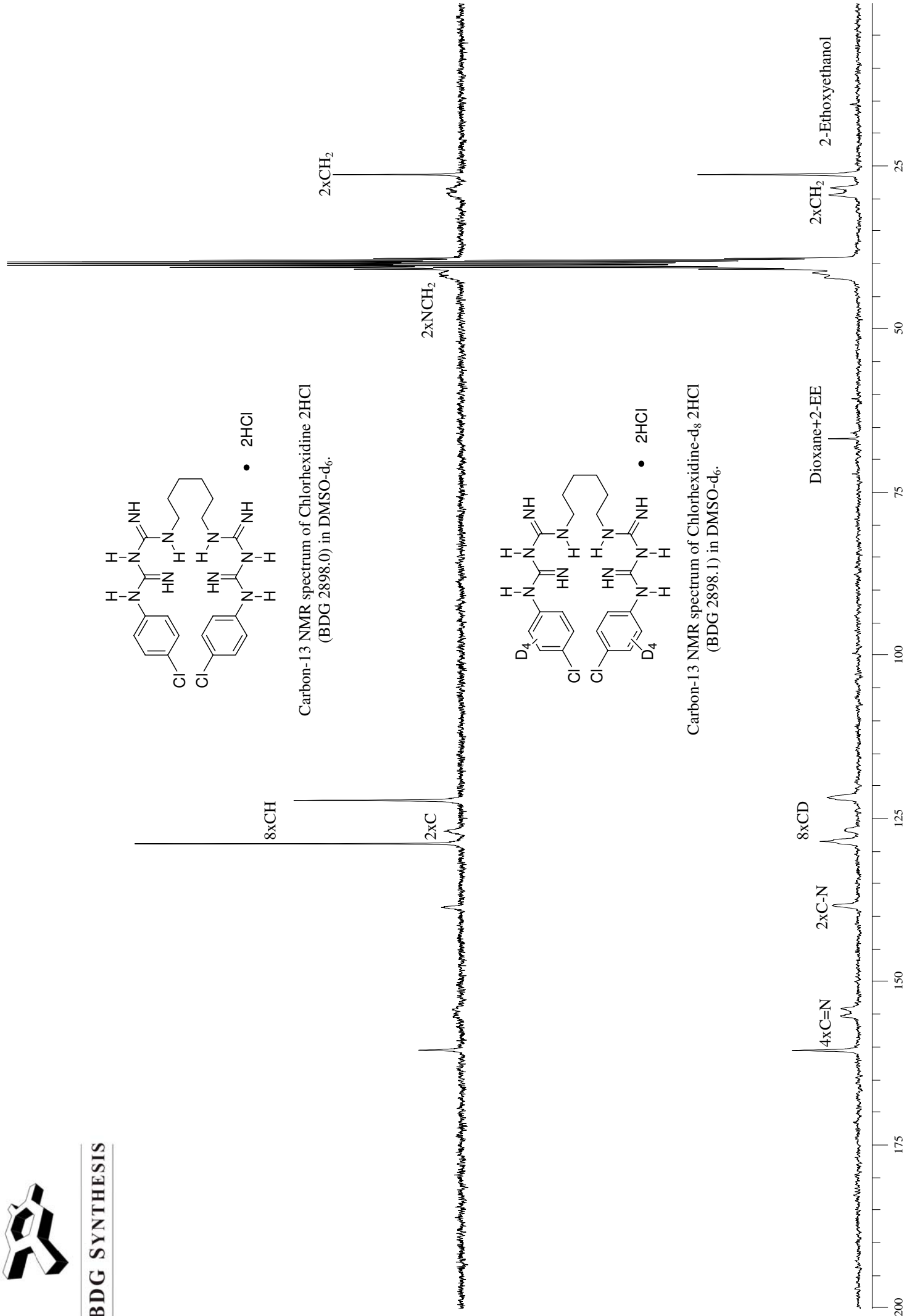




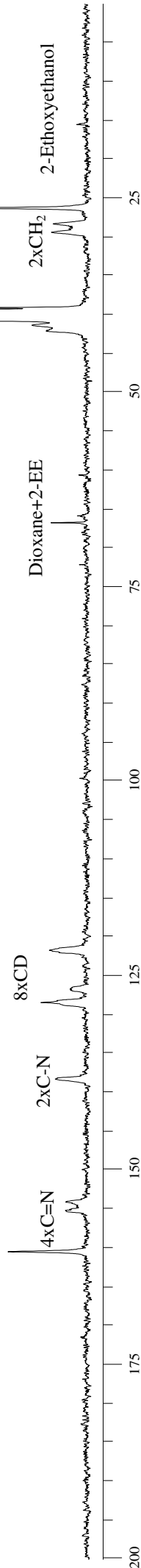
BDG SYNTHESIS



Carbon-13 NMR spectrum of Chlorhexidine 2HCl
(BDG 2898.0) in DMSO-d₆.



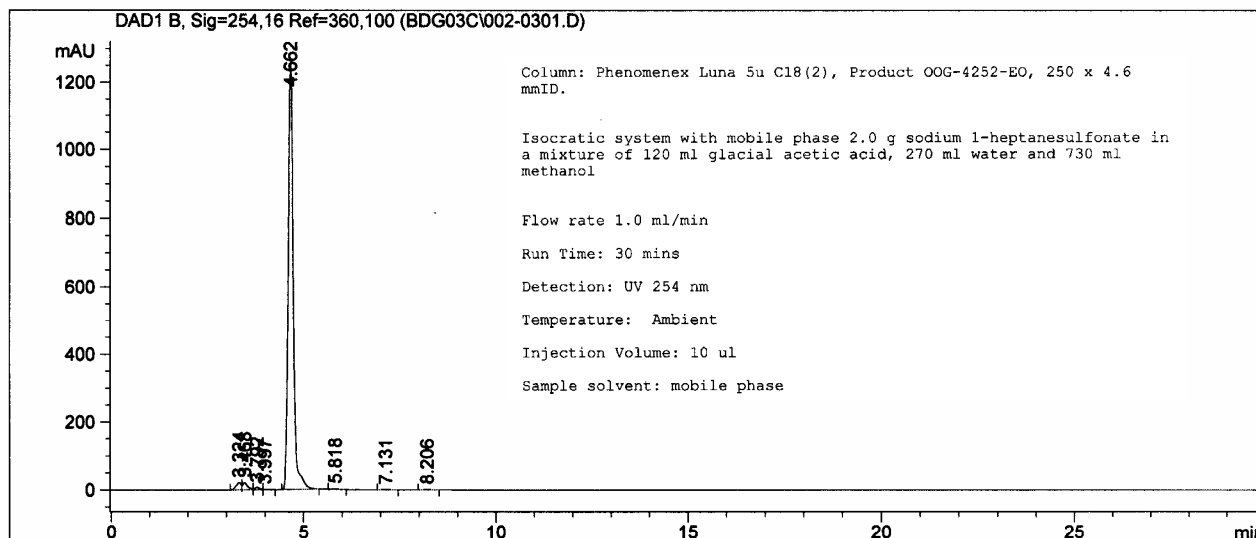
Carbon-13 NMR spectrum of Chlorhexidine-d₈ 2HCl
(BDG 2898.1) in DMSO-d₆.



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Injection Date   : 3/8/05 2:00:07 PM           Seq. Line :    3
Sample Name     : BDG 2898.1                 Location  : Vial 2
Acq. Operator  : YRLman                      Inj       :    1
                                           Inj Volume: 10 µl
Sequence File   : N:\LC1100_2\1\SEQUENCE\BDG03C.S
Method          : N:\LC1100_2\1\METHODS\LC40209A.M
Last changed   : 3/8/05 12:03:02 PM by YRLman
    
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Isocratic analysis of chlorhexidine on Luna C18 column # LC40209



Area Percent Report

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Sorted By      : Signal
Multiplier    : 1.0000
Dilution      : 1.0000
    
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Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.324	BV	0.1321	201.42892	22.15361	1.6774
2	3.468	VV	0.1288	201.21437	21.97148	1.6756
3	3.792	VV	0.1266	64.24012	7.60430	0.5350
4	3.997	VB	0.0929	8.53434	1.44586	0.0711
5	4.662	PB	0.1383	1.14797e4	1260.01160	95.5977
6	5.818	PP	0.1589	19.30656	1.92748	0.1608
7	7.131	PB	0.1921	18.39820	1.48944	0.1532
8	8.206	PP	0.1942	15.51355	1.22142	0.1292

Totals : 1.20083e4 1317.82519

Results obtained with enhanced integrator!

*** End of Report ***