Neogenkwanines A–H: Daphnane-Type Diterpenes Containing 4,7 or 4,6- Ether Group from the Flower Bud of Daphne genkwa

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1. HRESIMS spectrum of compound 1.
2. $^1$H NMR spectrum for compound 1 recorded in CDCl$_3$ at 600 MHz.
3. $^{13}$C NMR spectrum for compound 1 recorded in CDCl$_3$ at 125 MHz.
4. HMBC spectrum for compound 1 recorded in CDCl$_3$ at 600 MHz.
5. NOESY spectrum for compound 1 recorded in CDCl$_3$ at 600 MHz.
7. HRESIMS spectrum for compound 2.
8. $^1$H NMR spectrum for compound 2 recorded in CDCl$_3$ at 600 MHz.
9. $^{13}$C NMR spectrum for compound 2 recorded in CDCl$_3$ at 125 MHz.
10. HSQC spectrum for compound 2 recorded in CDCl$_3$ at 600 MHz.
11. HMBC spectrum for compound 2 recorded in CDCl$_3$ at 600 MHz.
12. NOESY spectrum for compound 2 recorded in CDCl$_3$ at 600 MHz.
13. HRESIMS spectrum for compound 3.
14. $^1$H NMR spectrum for compound 3 recorded in CDCl$_3$ at 600 MHz.
15. $^{13}$C NMR spectrum for compound 3 recorded in CDCl$_3$ at 125 MHz.
16. HMBC spectrum for compound 3 recorded in CDCl$_3$ at 600 MHz.
17. NOESY spectrum for compound 3 recorded in CDCl$_3$ at 600 MHz.
18. H-H-COSY spectrum for compound 3 recorded in CDCl$_3$ at 600 MHz.
20. $^1$H NMR spectrum for compound 4 recorded in CDCl$_3$ at 600 MHz.
21. $^{13}$C NMR spectrum for compound 4 recorded in CDCl$_3$ at 125 MHz.
22. HMBC spectrum for compound 4 recorded in CDCl$_3$ at 600 MHz.
23. HRESIMS spectrum for compound 5.
24. $^1$H NMR spectrum for compound 5 recorded in CDCl$_3$ at 600 MHz.
25. $^{13}$C NMR spectrum for compound 5 recorded in CDCl$_3$ at 125 MHz.
26. HMBC spectrum for compound 5 recorded in CDCl$_3$ at 600 MHz.
27. HRESIMS spectrum for compound 6.
28. $^1$H NMR spectrum for compound 6 recorded in CDCl$_3$ at 600 MHz.
29. $^{13}$C NMR spectrum for compound 6 recorded in CDCl$_3$ at 125 MHz.
30. HMBC spectrum for compound 6 recorded in CDCl$_3$ at 600 MHz.
31. HRESIMS spectrum for compound 7.
32. $^1$H NMR spectrum for compound 8 recorded in CDCl$_3$ at 600 MHz.
33. $^{13}$C NMR spectrum for compound 8 recorded in CDCl$_3$ at 125 MHz.
34. HSQC spectrum for compound 8 recorded in CDCl$_3$ at 600 MHz.
35. HMBC spectrum for compound 8 recorded in CDCl$_3$ at 600 MHz.
36. NOESY spectrum for compound 8 recorded in CDCl$_3$ at 600 MHz.
37. HRESIMS spectrum for compound 8.
38. $^1$H NMR spectrum for compound 8 recorded in CDCl$_3$ at 600 MHz.
39. $^{13}$C NMR spectrum for compound 8 recorded in CDCl$_3$ at 125 MHz.
40. HSQC spectrum for compound 8 recorded in CDCl$_3$ at 600 MHz.
41. HMBC spectrum for compound 8 recorded in CDCl$_3$ at 600 MHz.
42. NOESY spectrum for compound 8 recorded in CDCl$_3$ at 600 MHz.
43. HRESIMS spectrum for compound 9.
44. $^1$H NMR spectrum for compound 9 recorded in CDCl$_3$ at 600 MHz.
45. $^{13}$C NMR spectrum for compound 9 recorded in CDCl$_3$ at 125 MHz.
46. CD spectrum for compounds 1-8.
47. CD spectrum for the combination of several compounds.
1: HRESIMS spectrum of compound 1

Compound Mass Spectrum List Report

Analysis Info
Analysis Name: D:\data\songshaojiang\plotfiles\Y4.22.A.d
Method: tune_low.m
Sample Name: 3(o)-1.0h-MS
Comment:

Acquisition Date: 2000-7-7 17:53:38
Operator: Bruker Customer

Acquisition Parameter
Source Type: ESI
Ion Polarity: Positive
Focus: Active
Scan Begin: 100 m/z
Scan End: 1000 m/z
Scan Step: 0.01 m/z
Set Collision Cell RF: 120.0 Vpp
Set Diverter Valve: Source

Intens.:

m/z 127.2253

m/z 627.2263

Summary:

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</tbody>
</table>

Bruker Daltonics DataAnalysis 3.4
printed: 2000-7-11 12:53:30
$^2$H NMR spectrum for compound 1 recorded in CDCl$_3$ at 600 MHz.
$^{13}$C NMR spectrum for compound 1 recorded in CDCl$_3$ at 125 MHz.
4: HMBC spectrum for compound 1 recorded in CDCl₃ at 600 MHz.
S 5

5: NOESY spectrum for compound 1 recorded in CDCl₃ at 600 MHz.
6. Single crystal X-ray diffraction analysis of genkdaphnin A (1)

Crystallization of genkdaphnin A (1) from n-hexane/ethyl acetate gave flaky cocrystal (1: 1) of 1 and ethyl acetate. All measurements were made on a Bruker SMART APEX-II with RAPID diffractometer using filtered CuKα radiation (λ = 1.54187 Å). Crystal data: Formula C_{27}H_{36}O_{9}, Formula weight 504.24, Orthorhombic system, Space group: P 2_1 2_1 2_1, a = 11.7035(7) Å, b=12.7664(9) Å, c=21.1157(14) Å, α=β=γ=90.00°, V = 3154.9 (4) Å³, Z = 4, T = 296 (2) K, d = 1.248 g cm⁻³, specimen: 0.05×0.20×0.010 mm³. The total number of independent reflections measured was 9697, 4408 reflections unique, of which 4183 were observed (|F|^2 ≥ 2σ(F)^2). All calculations were performed using Crystal Structure except for refinement, which was performed using direct method SHELXL-97, expanded by using difference Fourier techniques, and refined by the full-matrix least-squares calculations. The non-hydrogen atoms were refined anisotropically, and hydrogen atoms were included at their calculated positions. The final indices were R₁ = 0.0429, wR₂ = 0.1181 (w = 1/σ(F)^2), S = 1.030. Crystallographic data for genkdaphnin A (1) have been deposited at the Shanghai Institute of Pharmaceutical Industry.

ORTEP drawing of genkdaphnin A (1).
7: HRESIMS spectrum for compound 2
$^1$H NMR spectrum for compound 2 recorded in CDCl$_3$ at 600 MHz.
$^{13}$C NMR spectrum for compound 2 recorded in CDCl$_3$ at 125 MHz.
HSQC spectrum for compound 2 recorded in CDCl₃ at 600 MHz.
11: HMBC spectrum for compound 2 recorded in CDCl₃ at 600 MHz.
12: NOESY spectrum for compound 2 recorded in CDCl₃ at 600 MHz.
13. HRESIMS spectrum for compound 3.
$^{1}{H}$ NMR spectrum for compound 3 recorded in CDCl$_3$ at 125 MHz.
$^{15}$C NMR spectrum for compound 3 recorded in CDCl$_3$ at 600 MHz.
HMBC spectrum for compound 3 recorded in CDCl₃ at 600 MHz.
17: NOESY spectrum for compound 3 recorded in CDCl₃ at 600 MHz.
18: H-HCOSY spectrum for compound 3 recorded in CDCl$_3$ at 600 MHz.
20. $^1$H NMR spectrum for compound 4 recorded in CDCl$_3$ at 600 MHz.
21. $^{13}$C NMR spectrum for compound 4 recorded in CDCl$_3$ at 125 MHz.
22. HMBC spectrum for compound 4 recorded in CDCl₃ at 600 MHz.
23. HR-ESI-MS spectrum for compound 5.
24. $^1$H NMR spectrum for compound 5 recorded in CDCl$_3$ at 600 MHz.
25. $^{13}$C NMR spectrum for compound 5 recorded in CDCl$_3$ at 125 MHz.
26. HMBC spectrum for compound 5 recorded in CDCl$_3$ at 600 MHz.
28. $^1$H NMR spectrum for compound 6 recorded in CDCl$_3$ at 600 MHz.
29. $^{13}$C NMR spectrum for compound 6 recorded in CDCl$_3$ at 125 MHz.
30. HMBC spectrum for compound 6 recorded in CDCl₃ at 600 MHz.
31. HRESIMS spectrum for compound 7.
32. $^1$H NMR spectrum for compound 8 recorded in CDCl$_3$ at 600 MHz.
33. $^{13}$C NMR spectrum for compound 8 recorded in CDCl$_3$ at 125 MHz.
34. HSQC spectrum for compound 8 recorded in CDCl₃ at 600 MHz.
HMBC spectrum for compound 8 recorded in CDCl₃ at 600 MHz.
36. NOESY spectrum for compound 8 recorded in CDCl₃ at 600 MHz.
Compound Mass Spectrum List Report

Acquisition Info
Analysis Name: Didau/compounds/anglejad/fabm/9-23.B.d
Acquisition Date: 2009-7-7 18:01:58
Method: None, ion mode
Sample Name: 39(5)-15.9-MIS
Operator: None, Ionizer
Instrument: ESI, microOTOF-Q 125

Acquisition Parameters
- Spectroscopy: ESI
- Quadrupole: Q1
- Mass Range: 260-2600
- Start Value: 100 m/z
- End Value: 2000 m/z
- Resolution: 200000
- Scan Time: 5s

Spectrum for compound 8.
38. $^1$H NMR spectrum for compound 8 recorded in CDCl₃ at 600 MHz.
39. $^{13}$C NMR spectrum for compound 8 recorded in CDCl$_3$ at 125 MHz.
40. HSQC spectrum for compound 8 recorded in CDCl₃ at 600 MHz.
41. HMBC spectrum for compound 8 recorded in CDCl₃ at 600 MHz.
42. NOESY spectrum for compound 8 recorded in CDCl₃ at 600 MHz.
HRESIMS spectrum for compound 9.
44. $^1$H NMR spectrum for compound 9 recorded in CDCl$_3$ at 600 MHz.
$^{13}$C NMR spectrum for compound 9 recorded in CDCl$_3$ at 125 MHz.
46. CD spectrum for compounds 1-8.

CD spectrum for compound 1. CD spectrum for compound 2.

CD spectrum for compound 5. CD spectrum for compound 6.

CD spectrum for compound 7. CD spectrum for compound 8.
47. CD spectrum for the combination of several compounds
CD spectrum for compounds 1, 2 and 8

CD spectrum for compounds 3 and 4
CD spectrum for compounds 5 and 6

CD spectrum for compounds 8 and 8
48. Stereoviews of compounds 1-8