Terpene cyclopropyl alcohol

<table>
<thead>
<tr>
<th>Chemical Formula</th>
<th>Molecular Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>C_{20}H_{30}O_{2}</td>
<td>302.45 (MS has loss of H2O)</td>
</tr>
</tbody>
</table>

Paramuriceidae 24-VIII-85-1-006 SAM 33-82-01

No publication

PO NUMBER: PO40657  Bar Code: 1447414

\( \text{hplc (trace attached)} \)

Method: Vydac C18 Protein and Peptide, 4.6 x 250mm, 10\( \mu \)
flow 1 ml/min, detection: PDA: UV (extracted at 220 nm, black), ELSD (green dotted)
A: H\textsubscript{2}O:CH\textsubscript{3}CN (95:5, v/v), B: CH\textsubscript{3}CN
t=0 min A:B (90:10, v/v), t=20 min (100%B), t=28 min (100%B)

\( \text{LC-MS (spectrum attached) we see loss of H2O- but NMR is consistent with this assignment} \)

Method: Vydac C18 Protein and Peptide, 2.1x150 mm, flow 0.2 ml/min
A: H\textsubscript{2}O (0.1% formic acid), B: CH\textsubscript{3}CN (0.1% formic acid)
t= 0 min A:B (90:10, v/v), t=15 min (100%B), t=21 min (100%B), t=22.1 min A:B(90:10, v/v)
using a linear gradient

\( ^{1}H \text{ (600 MHz)} \)

\( \text{CDCl}_3 \) \( \square \) \( \text{CD}_3\text{OD (Methanol-d}_4 \) \( \square \) \( \text{CDCl}_3/\text{CD}_3\text{OD} \)

\( ^{13}\text{C} \text{ (150 MHz)} \)

\( \text{CDCl}_3 \) \( \square \) \( \text{CD}_3\text{OD (Methanol-d}_4 \) \( \square \) \( \text{CDCl}_3/\text{CD}_3\text{OD} \)

\( \text{solubility} \)

\( \text{CHCl}_3/\text{MeOH (9:1)} \) \( \square \) \( \text{MeOH} \) \( \square \) \( \text{DMSO} \)

\( \text{estimated purity} \geq 90\% \)

\( \text{sample weight} \geq 10.5 \text{ mg} \)

For further information contact:  \hspace{1cm}  \text{Date:__________}  
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awrich33@hboi.fau.edu, 772-242-2459
24-VIII-85-1-006
Orange sea fan
103g extd/255g left
Dionex extraction
2 cells
3 static cycles per step

24-VIII-85-1-006
Orange sea fan
sample was mainly stalks
169g/85g left
Dionex extraction
2 cells 3 steps at 100C
3 static cycles per step

Sam27-57-01
Heptane extract
1.69g
Sam27-57-02
EtOH:EtOAc 5:1
extract
1g
Sam27-57-01 combined with -02. 1.19g
run on a 30g C18 RP Rf Gold column.
H2O 75% to ACN grad., MeOH to DCM
grad., TFA wash

Sam30-99-01
Heptane extract
765 mg
Sam30-99-02
EtOH:EtOAc 5:1
extract
421 mg
Sam30-99-01 combined with -02. 1.19g
run on a 30g C18 RP Rf Gold column. H2O
75% to ACN grad., MeOH to DCM
grad., TFA wash

Remainders of samples Sam30-86-08 (13.4mg), Sam30-99-08 (20mg) and all of Sam33-60-04 thru 07 (12mg) were further purified via Semi prep C18, 30 min. 60:40 A:B isocratic- A= 85:5 H2O:ACN B=ACN

RobL25-92-03 and Sam33-63-03 combined to make
Sam33-73-01
This was further purified via Semi prep C18 60:40 iso for 20 min, 100% B by 20 min, hold for 3 min, 30:70 by 30 min. A= 95:5 H2O:ACN B=ACN

Sam33-71-01 and Sam33-73-03 determined to be the same(!) and combined to make
Sam33-82-01 (HB371) 17.9mg

RobL25-92-03 and Sam33-63-03 combined to make
Sam33-73-01
This was further purified via Semi prep C18 60:40 iso for 20 min, 100% B by 20 min, hold for 3 min, 30:70 by 30 min. A= 95:5 H2O:ACN B=ACN
D-2000 Elite HPLC System Manager Report

Analyzed: 01/08/2014 12:20 PM  
Reported: 01/08/2014 02:42 PM

Sample Name: Sam33-82-01 HB371
Sample Description: 24-VIII-85-1-006 HB371

 Acquisition Method: autosampler_30min_wELSD
 Column Type: Vydac C18
 Pump A Solvent A: H2O/5% ACN
 Pump A Solvent B: ACN