

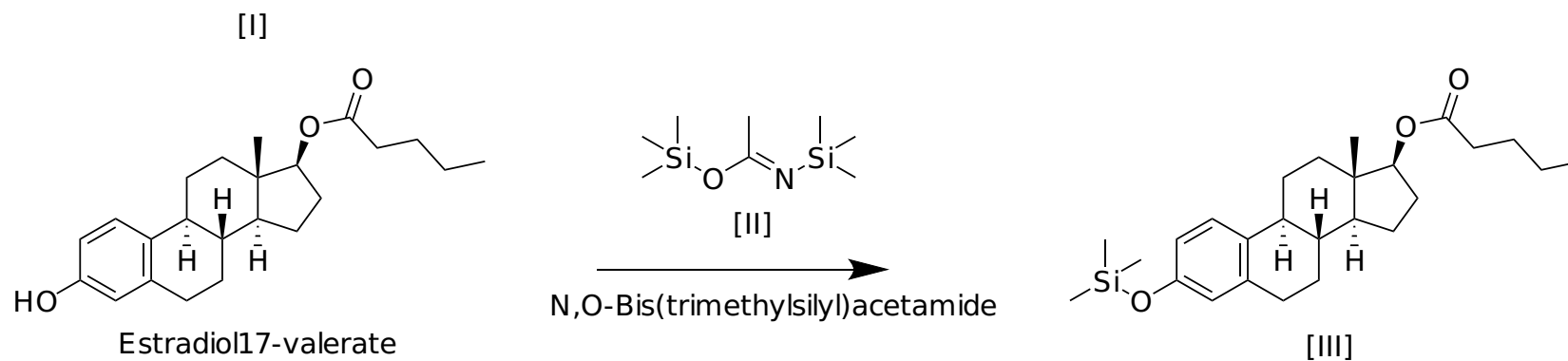
Mixed Solvent Silylation & Further GCMS Method Development

| | |
|-----------------|---|
| Notebook | openEst |
| Concept | |
| Labhead | |
| Program | |
| Owner | Gwyn Uttmark uttmark@stanford.edu |
| Created | Gwyn Uttmark uttmark@stanford.edu Jan 29, 2020 12:53 PM PST |
| Modified | Gwyn Uttmark uttmark@stanford.edu Feb 1, 2020 9:39 PM PST |

Status: Active

Content

Scheme



Reactants

| Rxn ID | Reactant | MF | FM | MW | EM | Limit? | Eq |
|--------|---|--|--------------|--------------|-----------|--------|----|
| I | (8R,9S,13S,14S,17S)-3-hydroxy-13-methyl-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-17-yl pentanoate | C ₂₃ H ₃₂ O ₃ | 356.51 g/mol | 356.51 g/mol | 356.23514 | ✓ | 1 |
| II | trimethylsilyl (E)-N-(trimethylsilyl)acetimidate | C ₈ H ₂₁ NO ₅ Si ₂ | 203.43 g/mol | 203.43 g/mol | 203.11617 | | 1 |

Products

| Rxn ID | Product ID | Product | MF | FM | MW | EM |
|--------|------------|---|---|--------------|--------------|-----------|
| III | P1 | (8R,9S,13S,14S,17S)-13-methyl-3-((trimethylsilyloxy)-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-17-yl) pentanoate | C ₂₆ H ₄₀ O ₃ Si | 428.69 g/mol | 428.69 g/mol | 428.27467 |

Prelab Outline

Reagent Table:

| Chemical | Equivalent | Weight/Volume | Mole Quantities (umol) | MW (g/mol) | Density (g/mL // weqd) | BP/MP (C) |
|----------|------------|---------------|------------------------|------------|------------------------|-----------|
| Methanol | 4400 | Varied | 24720/mL | 32.04 | 0.792 | 65 |

| | | | | | | |
|---------------------------------------|------|----------------|----------|---------|-------|-----|
| Acetonitrile | 3400 | Varied | 19070/mL | 41.05 | 0.783 | 82 |
| Estradiol Valerate | 1 | 2mg/per sample | 5.61 | 356.498 | N/A | N/A |
| bis(trimethylsilyl)acetamide (TMS-BA) | 146 | 0.2mL | 818 | 203.43 | 0.832 | 71 |

Sample Table:

| Sample(s) | mL AcN | mL Methanol | mg EV | mL TMS-BA |
|-----------|--------|-------------|-------|-----------|
| 1A, 1B | 1 | 5 | 2 | 0.2 |
| 2A, 2B | 2 | 4 | 2 | 0.2 |
| 3A, 3B | 3 | 3 | 2 | 0.2 |
| 4A, 4B | 4 | 2 | 2 | 0.2 |
| 5A, 5B | 5 | 1 | 2 | 0.2 |

1. Make 2mg/mL AcN solution (40mg/20mL)
 1. 0.0407g EV added to 2*10ml vflask acn
 2. vortexed to mix
2. Pipette 1mL of EV-AcN solution into 8mL vials
3. Pipette 0.2mL TMS-BA into vial and cap
4. Sonicate at ~30-35C for 15min
5. Cool while covered in an ice bath
6. Add 1mL MeOH solution dropwise
 1. The solution did not grow appreciably warm during this step, even when sample was removed from ice bath before addition
7. Add rest of AcN solution (variable based on sample)
8. Add rest of MeOH solution (variable based on sample)
9. Prep whattman filtered samples (for a total of 10 final samples)

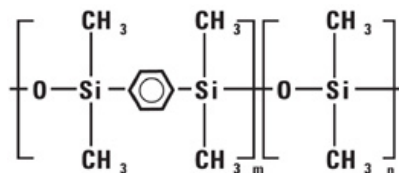
GCMS:

1. Re-run:
 1. Oct21
 2. Poster
 3. 260mt
2. Modify
 1. Poster (to make it shorter)

2. A mid-point between 260mt and Poster
3. Upload profile image

GCMS Notes

Column: <https://www.agilent.com/en/product/gc-columns/high-temperature-gc-columns/db-5ht-columns>



DB-5HT Column (5% phenyl) methylpolysiloxane

1-5:1A-5A

6-10:1B-5B

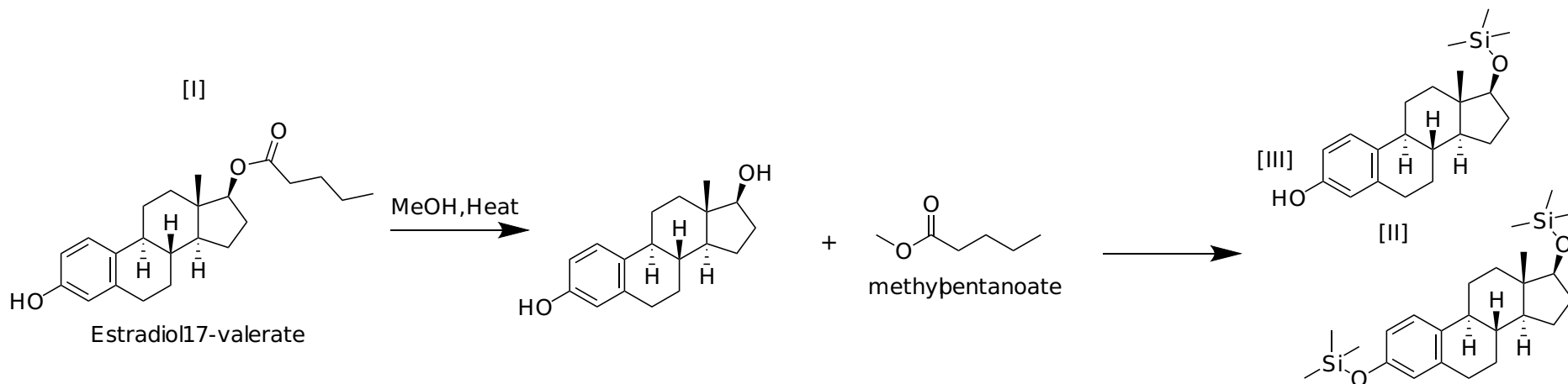
11: Methanol Blank

Initial run halted because all the blanks were named the same thing. Listen to error messages!!

seq-jan31.PNG

| | | | | | | | |
|----|---------------------|----|----------------------------|------------------------|----------------------------|---------------------|--------|
| 1 | blank1 | 11 | D:\est\biome | nov4-blanking.M | D:\est\biome\jan31-data\1a | blank1 | Blank |
| 2 | oct21 | 1 | D:\est\biome | oct21.M | D:\est\biome\jan31-data\1a | oct21 | Sample |
| 3 | blank2 | 11 | D:\est\biome | nov4-blanking.M | D:\est\biome\jan31-data\1a | blank1 | Blank |
| 4 | poster | 1 | D:\est\biome\jan13-methods | poster-replicate.M | D:\est\biome\jan31-data\1a | poster | Sample |
| 5 | blank3 | 11 | D:\est\biome | nov4-blanking.M | D:\est\biome\jan31-data\1a | blank1 | Blank |
| 6 | 260mt | 1 | D:\est\biome | 260mt.M | D:\est\biome\jan31-data\1a | 260mt | Sample |
| 7 | blank4 | 11 | D:\est\biome | nov4-blanking.M | D:\est\biome\jan31-data\1a | blank1 | Blank |
| 8 | posterShortEdit | 1 | D:\est\biome\jan31-methods | poster-shortedit.M | D:\est\biome\jan31-data\1a | posterShortEdit | Sample |
| 9 | blank5 | 11 | D:\est\biome | nov4-blanking.M | D:\est\biome\jan31-data\1a | blank1 | Blank |
| 10 | posterVeryShortEdit | 1 | D:\est\biome\jan31-methods | poster-veryshortedit.M | D:\est\biome\jan31-data\1a | posterVeryShortEdit | Sample |
| 11 | blank1 | 11 | D:\est\biome | nov4-blanking.M | D:\est\biome\jan31-data\2a | blank1 | Blank |
| 12 | oct21 | 2 | D:\est\biome | oct21.M | D:\est\biome\jan31-data\2a | oct21 | Sample |

ChemDraw Document



Reactants

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| III | P2 | (((8R,9S,13S,14S,17S)-13-methyl-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthrene-3,17-diyl)bis(oxy))bis(trimethylsilane) | C ₂₄ H ₄₀ O ₂ Si ₂ | 416.75 g/mol | 416.75 g/mol | 416.25668 |

Solvents

| Solvent |
|----------|
| Methanol |

Reaction Conditions

| Pressure | Temperature | Reaction Time |
|----------|-------------|---------------|
| 1 atm | 30 °C | 15 min |

ChemDraw Document-1