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Catalytic synthesis of enantiopure mixed diacylglycerols - synthesis of a major M. tuberculosis phospholipid and platelet activating factor

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Catalytic synthesis of enantiopure mixed diacylglycerols – synthesis of a major *M. tuberculosis* phospholipid and platelet activating factor

Peter Fodran, A.J. Minnaard

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General information:

Material:

Solvents for chemical reactions were dried according to the standard procedures. Solvents for flash chromatography were used without further purification. All reagents were obtained from commercial suppliers and were used without further purification unless noted otherwise.

Methods:

All the reactions were performed using Schlenk techniques. Glassware was dried by heating (150°C) for at least 2 h and subsequent cooling under vacuum before use.

Reactions were monitored by GC/MS (GC, HP6890: MS HP5973) equipped with an HP1 column (Agilent Technologies, Palo Alto, CA) or by TLC on silica coated aluminium foils (60 Å, 0.25mm coating thickness). TLCs were visualized by the following stains: iodine stain, Seebach's stain, bromocresol green stain or ninhydrine (for phospholipids)

Flash column chromatography was performed on 230-430 mesh silica gel.

^1H , ^{13}C , ^{31}P NMR spectroscopy was recorded on Varian VXR300 or AMX400 spectrometers. Chemical shifts were determined relative to the residual solvent peaks (CHCl_3 , $\delta = 7.26$ ppm for ^1H NMR, $\delta = 77.0$ for ^{13}C NMR).

The (–) sign stands for negative phase in APT (Attached Proton test)

Optical rotations were measured on a Schmidt+Haensch polarimeter (Polartronic MH8) with a 10 cm cell.

The mass spectra were recorded on an Thermoscientific LTQ OrbitrapXL spectrometer.

NMR Spectra Data for synthesized compounds:

(E)-S-ethyl dec-2-enethioate (18)

(R)-S-ethyl 3-methyldecanethioate (19)

(R)-3-methyldecanal

(R)-10-methyloctadecenoic acid (20)

(R)-Tuberculostearic acid (21)

3-((tert-butyldimethylsilyl)oxy)-2-(palmitoyloxy)propyl stearate (12)

(S)-3-hydroxy-2-(palmitoyloxy)propyl stearate

(R)-(S)-3-hydroxy-2-(palmitoyloxy)propyl tuberculostearate

MTB lipid (22)

MTB lipid desmethyl analogue (13)

MTB lipid desmethyl analogue (14)

PAF precursor 26

PAF precursor 24

PAF precursor after desilylation

Spectrum contains acetone at 2.17 (NMR tube)

TBDPS-(R)-glycidyl ether

HPLC traces of ring-opened products:

Racemic:

<Peak Table>

AD2

Peak#	Ret. Time	Area	Height	Area%
Total				

PDA Ch1 190nm

Peak#	Ret. Time	Area	Height	Area%
1	9.868	33261044	2288284	56.680
2	11.240	25420716	1577430	43.320
Total		58681760	3865714	100.000

Optically pure

<Peak Table>

AD2

Peak#	Ret. Time	Height	Area	Area%
Total				

PDA Ch1 190nm

Peak#	Ret. Time	Height	Area	Area%
1	10.033	1526780	21064237	96.743
2	12.924	28991	709075	3.257
Total		1555771	21773312	100.000