Electronic Supplementary Material (ESI) for Analytical Methods.

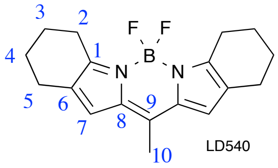
Electronic Supplementary Information

Synthesis and Characterisation of LD 540

Prepared based on report by Spandl, J.;White, D.J.; Peychl, J.; Thiele, C., *Traffic* **2009**, 10, 1579 and in US patent by Morgan L.R; Boyer, J.H. 446,157 1995

1 g of 4,5,6,7-tetrahydroindole was dissolved in 6 mL dry dichloromethane (CH_2Cl_2), and 1.6 mL of freshly distilled acetyl chloride was added dropwise followed by 0.4 mL of non-distilled acetyl chloride dropwise with stirring under nitrogen. The solution was refluxed for 1 h, then about half the dichloromethane was distilled at atmospheric pressure while the remainder was removed under reduced pressure (rotavap) leaving a gummy solid. This was triturated with 3 x 10 mL hexanes and then filtered. The solid material on the filter and remaining in the flask was dissolved in 100 mL dry CH_2Cl_2 and placed under nitrogen in a 250 mL RB flask with a septum and magnetic stirrer bar. Then 2.5 mL triethylamine and 2.5 mL BF_3.etherate were added separately alternately dropwise and the solution was stirred for 2 h under nitrogen. The solution was then stored 2 days under nitrogen prior to workup. The solution was evaporated almost to dryness (rotavap), then the residue was dissolved in CH_2Cl_2: hexanes 2:3 and loaded on a silica gel column prepared in the same solvent mixture. Then CH_2Cl_2 2:3 was used to elute a purple band that was discarded and then a strongly fluorescent red band. The eluate was taken almost to dryness, then a small volume of hexanes was added and the resulting solid was filtered and washed with hexanes (0.195 g, F1). A further 50 mg of less pure material was obtained by combining the filtrate and washings, evaporating (rotavap and then high vacuum), then triturating with a mix of hexanes and isopropanol and filtering. NMR, elemental analysis, and mass spectral measurements showed that the material in F1 was contaminated with triethylamine. This was removed by dissolving the solid in CH_2Cl_2, washing with dilute potassium dihydrogen citrate, then drying the solution with Na_2SO_4, adding hexanes, and evaporating the solution almost to dryness. The solid was filtered and washed with a small volume of hexane. Elemental analysis: Calculated for $C_{18}H_{21}NF_2N_2$ C: 68.8%, H: 6.74%, N: 8.92%. Found: C:68.3%, H: 6.80%, N: 8.71%. Mass spectrum (electrospray introduction, positive ion mode): (M+Na): 337.1673 Calculated for $C_{18}H_{21}BF_2NaN_2$: 337.1661

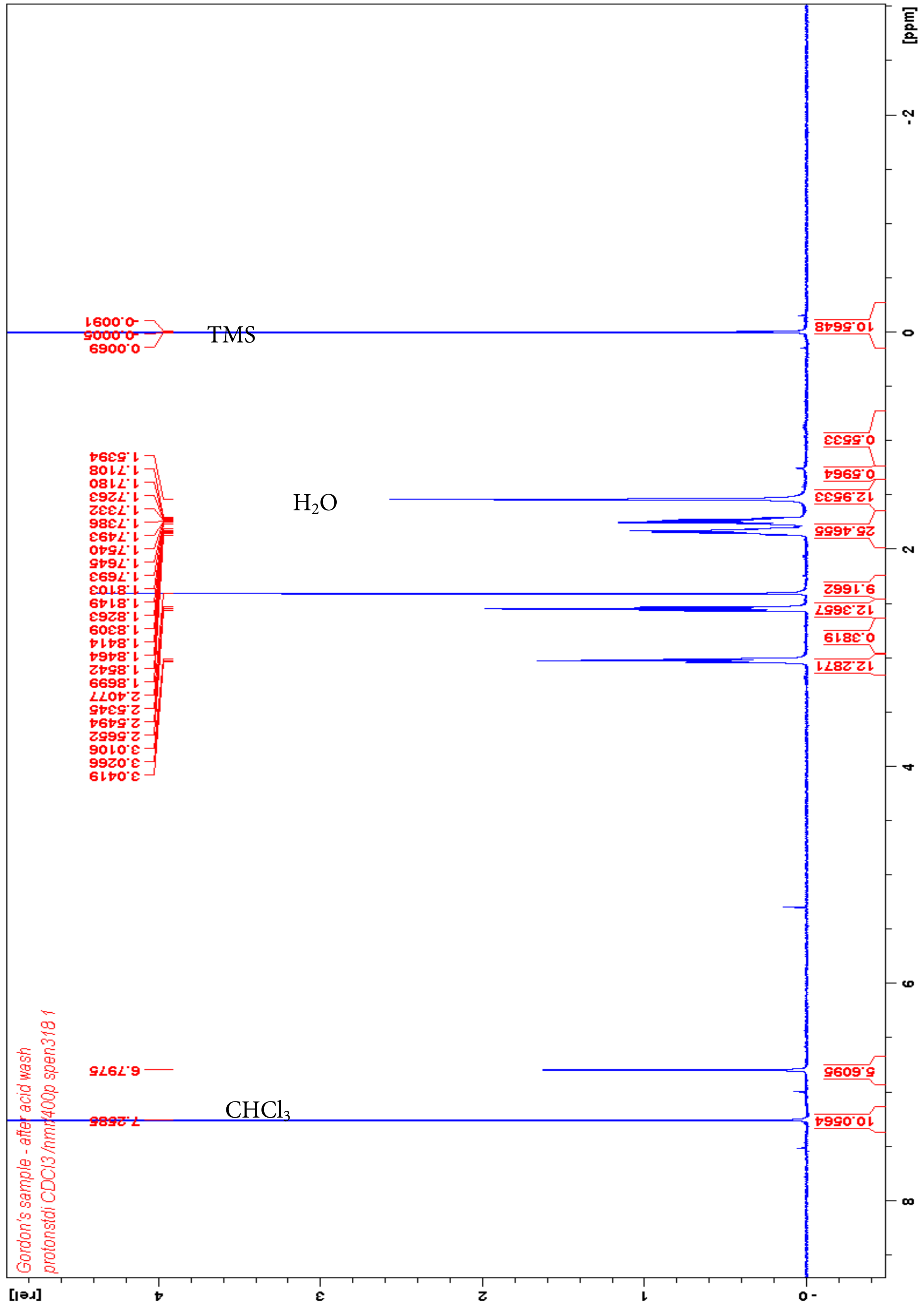
^{1}H NMR ($CDCl_3$): H(2) 3.02 (triplet, 4H) H(3) (or H(4)) 1.74 (multiplet, 4H), H(3) (or H(4)) 1.84 (multiplet, 4H), H(5) 2.55 (triplet, 4H), H(7) 6.80 (singlet, 2H), H(10) 2.40 (singlet, 3H); ^{13}C NMR ($CDCl_3$) C(1) 128.5, C(2) 24.5, C(3) (or C(5)) 23.2, C(4) 22.4, C(5) (or C(3)) 24.5, C(6) 156.4 , C(7) 123.1, C(8) 134.2, C(9) 138.4, C(10) 15.2

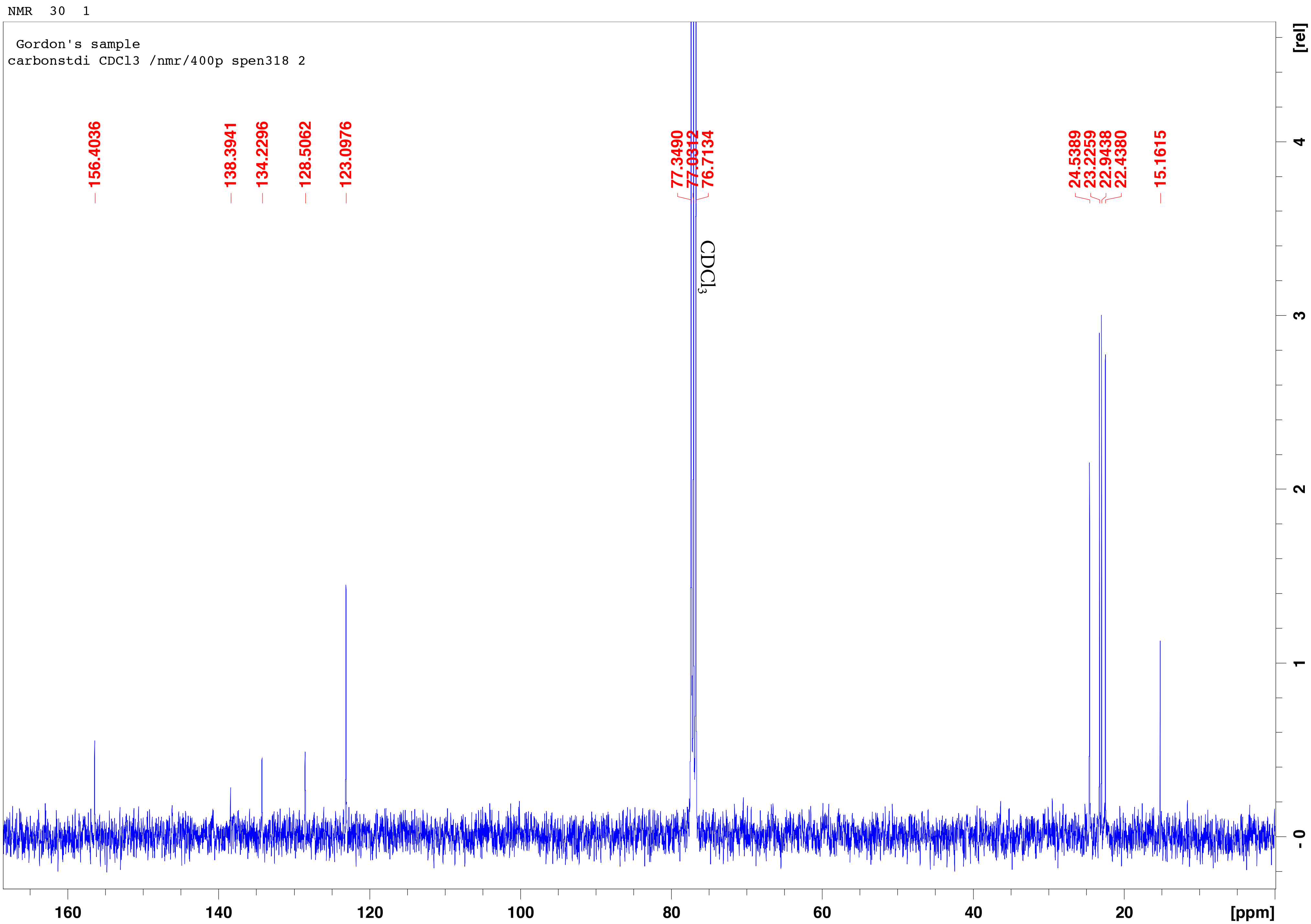


LD540

LD 540 after chromatography and acid wash

^{1}H NMR, decoupled ^{13}C NMR, HMBC, HSQC

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Gordon's sample
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77.0312
76.7134
CDCl$_3$
24.5389
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22.9438
22.4380
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20
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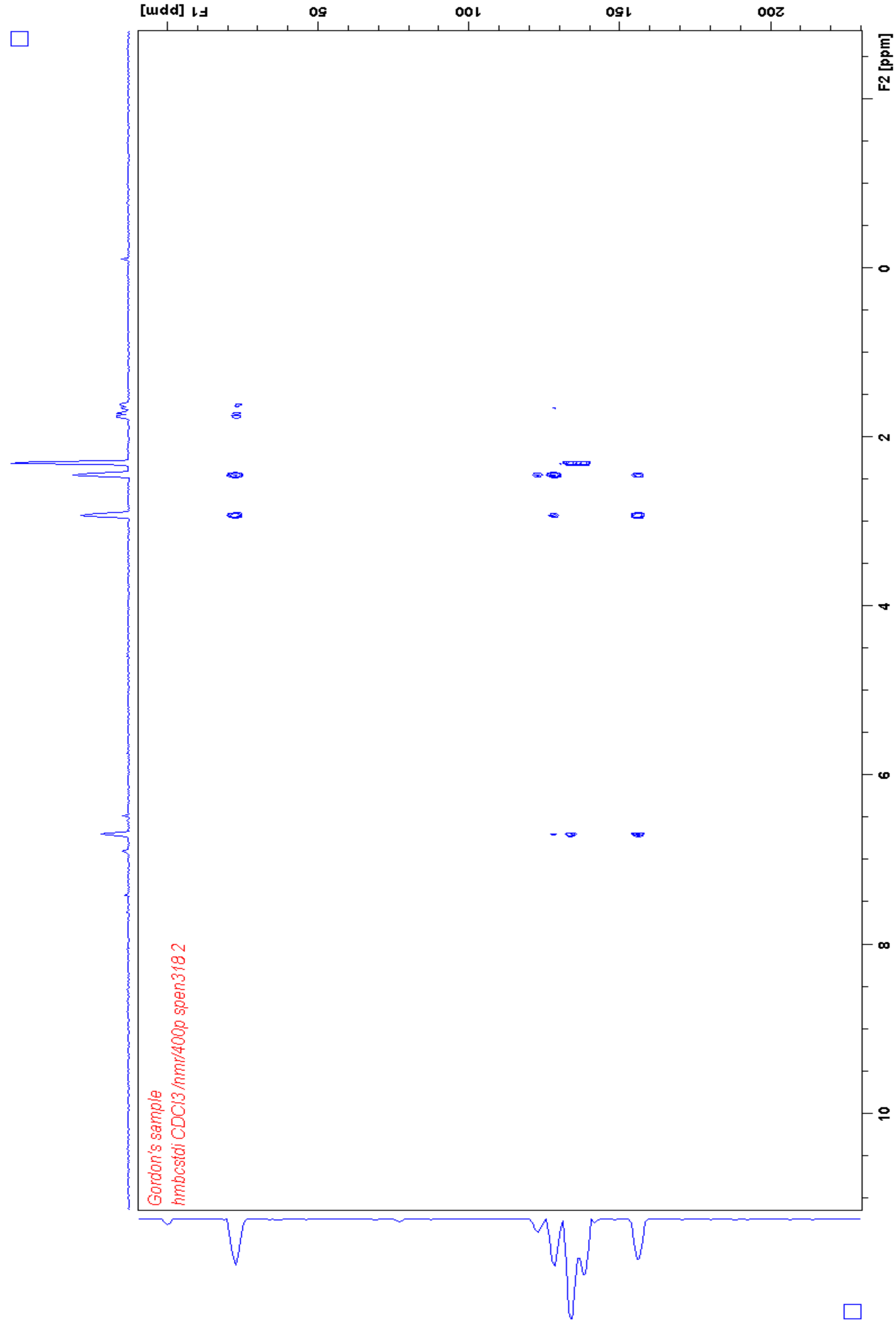
HMBC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8
10
Gordon's sample
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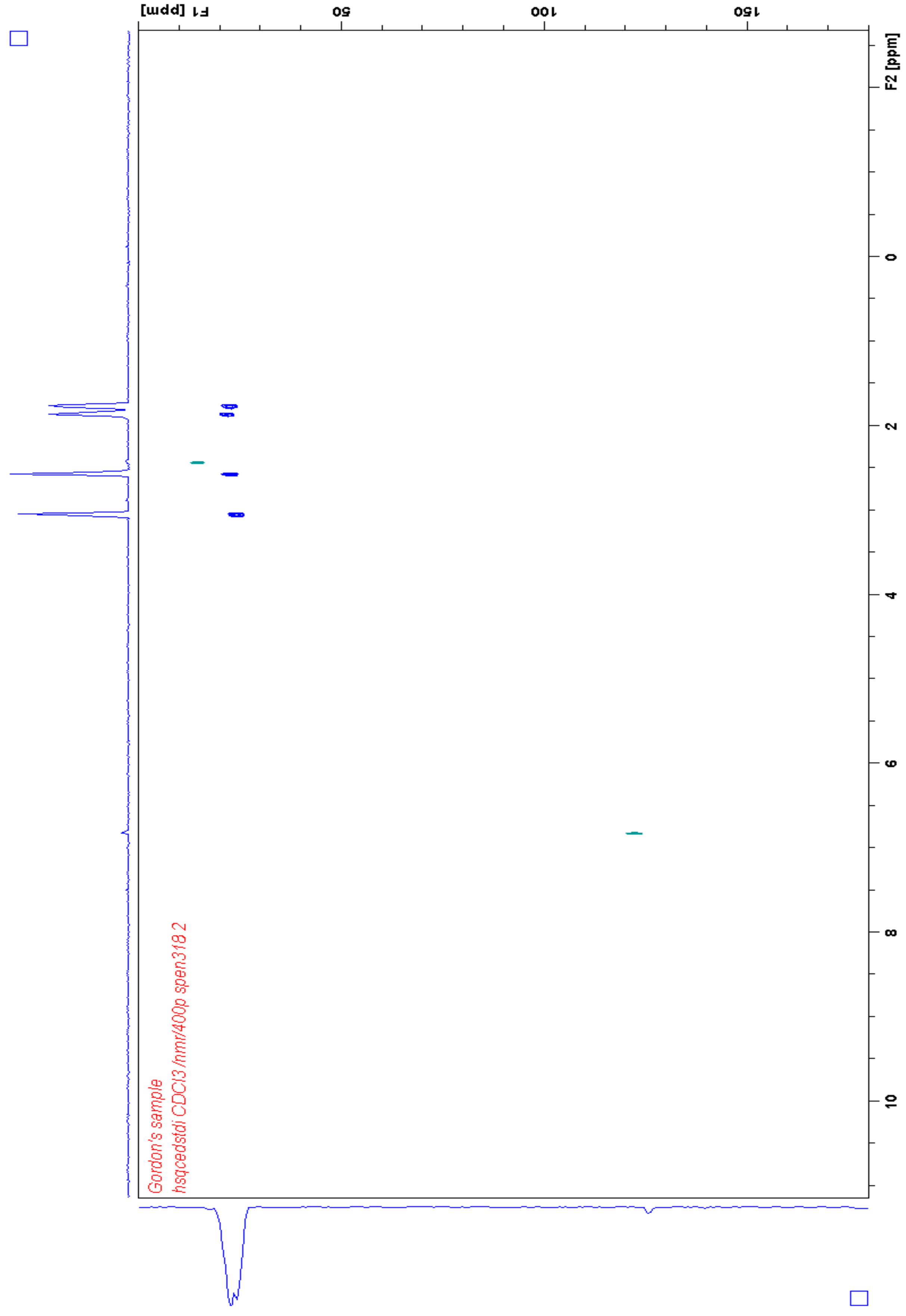
HSQC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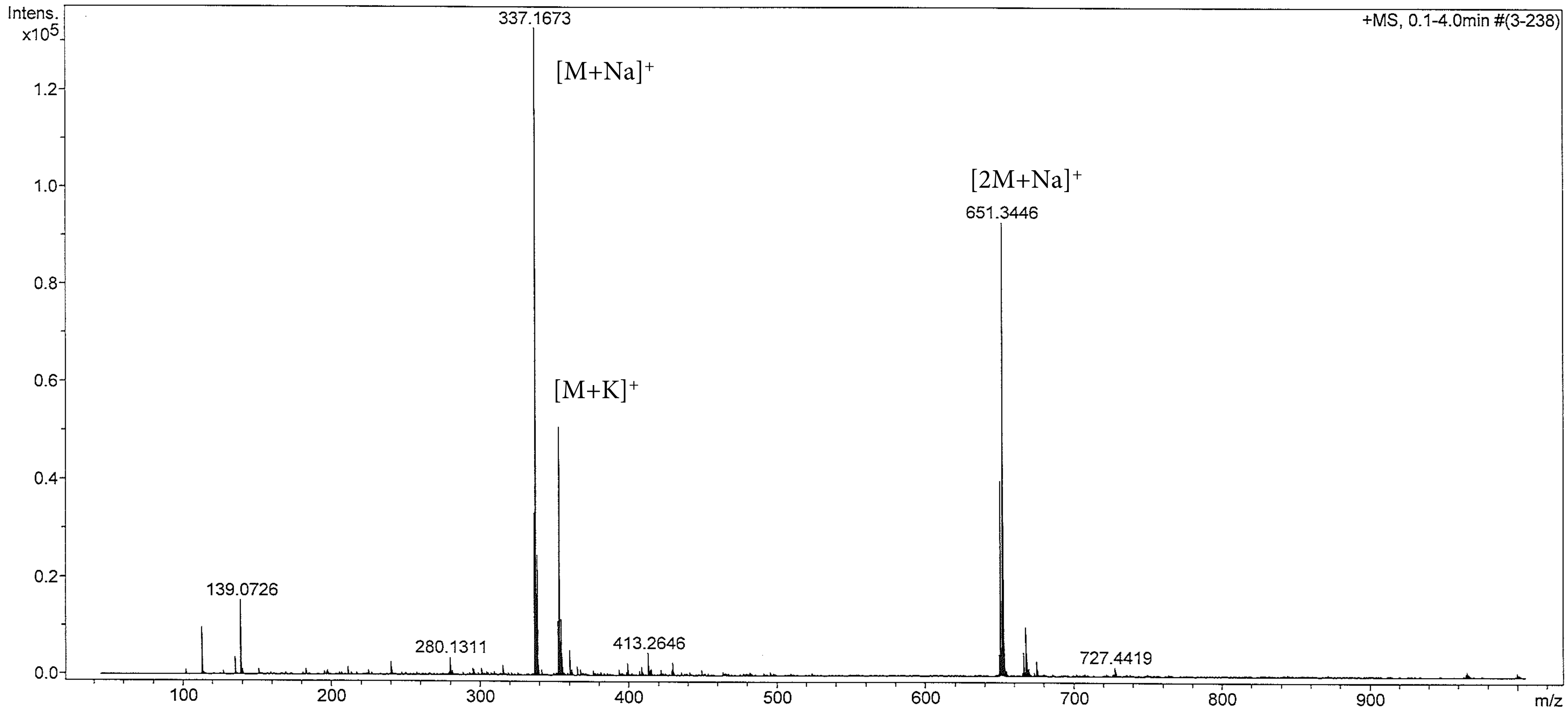
Generic Display Report

Analysis Info

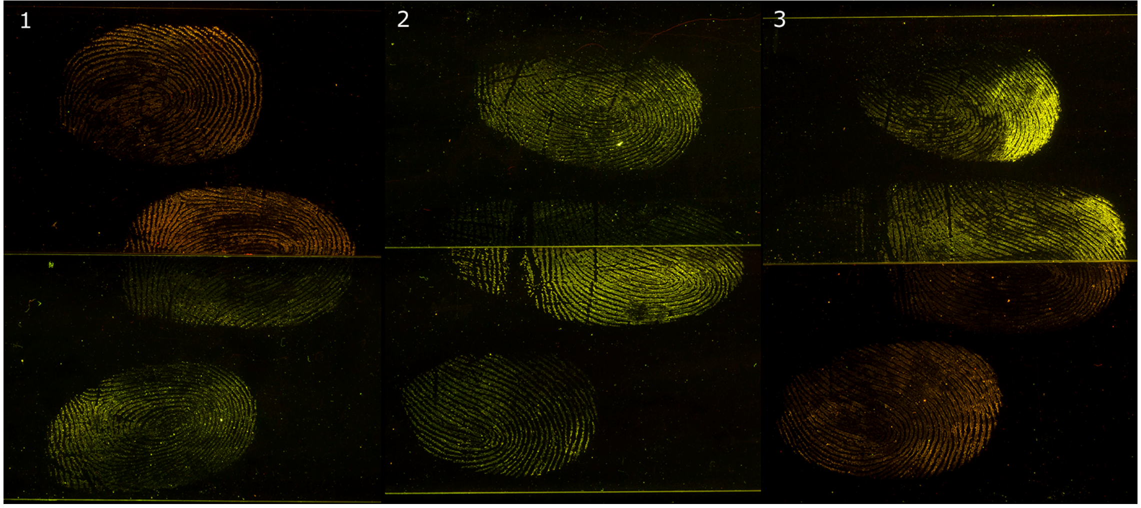
Analysis Name	D:\Data\Akld University\Nick Oct 12\12-10-18\Gordon000001.d
Method	120725 - Nick Positive 50-1000.m
Sample Name	GM1206
Comment	Dissolved sample in 1mL Acetone Diluted sample 2uL in 1mL MeOH

Acquisition Date	18/10/2012 11:06:57 a.m.
Operator	Nick
Instrument	micrOTOF-Q



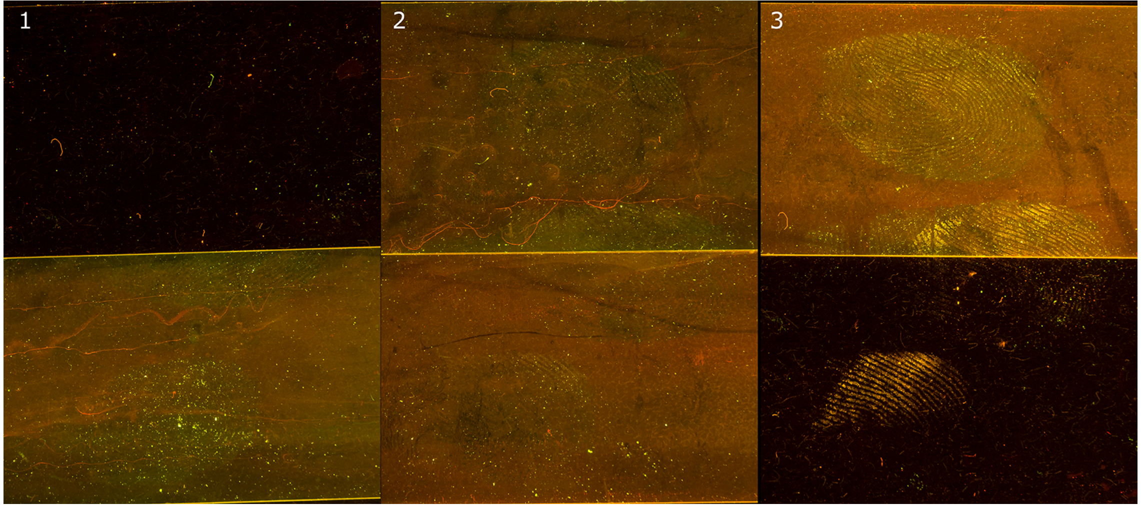
Enhancement of fingermarks on glass slides.
All fingermarks were 24 h old, and were either charged (by wiping fingers across the forehead) or uncharged (hands were washed 2 h prior to fingermark application, followedby normal activities. The methanolic aqueous Nile Red solution was prepared as described in M. de la Hunty X. Spindler, S. Chadwick, C. Lennard & C.. Roux, For. Sci. Int., 2014, 244, e48-e55 , but on a 1/10 scale. An LD 540 stock solution was prepared by dissolving 10 mg LD540 in 50 mL HFE-7100. This was then diluted 1:1 with either HFE-7100 or with perfluorooctane. Fingermarks were exposed to the solutions for 5 min, and were then rinsed with water (Nile Red) or HFE-7100 or 1:1 HFE-7100: perfluorooctane.
Illumination for the fluorescent imaging was from a Polilight PL10 with excitation band centred at 505 nm. Images were taken with a Canon EOS 550D with 100 mm macro lens and KV550 filter, using DSLR remote (Breeze Systems) with manual control, ASA 400, “tungsten” white balance setting, with images stored both as jpg and cr2 (Raw). Images shown below are from cr2 images with no changes in brightness or contrast.

Charged fingermarks on glass slides. Images taken with exposure time of 4s and aperture = 11



1 Upper: Nile red in aqueous methanol Lower: LD540 in HFE7100
2 Upper: LD540 in HFE7100 Lower: LD540 in HFE7100:perfluorooctane 1:1
3 Upper: LD540 in HFE 7100:perfluorooctane 1:1 Lower Nile red in aqueous methanol

Uncharged fingermarks on glass slides. Images taken with exposure times of 4 s and aperture 2.8.



1 Upper: Nile red in aqueous methanol Lower: LD540 in HFE7100
2 Upper: LD540 in HFE7100 Lower: LD540 in HFE7100:perfluorooctane 1:1
3 Upper: LD540 in HFE 7100:perfluorooctane 1:1 Lower Nile red in aqueous methanol

Imaging of sebaceous fingermarks on ice

Sebaceous fingermarks were deposited on ice at -15 °C and then stored at -15 °C for the times shown until development. Enhancement was with a saturated solution of LD540 in HFE-7100:perfluorooctane 1:1 at -15 °C. Imaging was performed as described previously, except the sample was placed on a platform in an insulated container containing dry ice. The temperature at the sample during imaging was approximately -35 °C.