

Synthesis and Radiosynthesis of a Novel PET Fluorobenzyl Piperazine for Melanoma Tumour Imaging; [¹⁸F]MEL054.

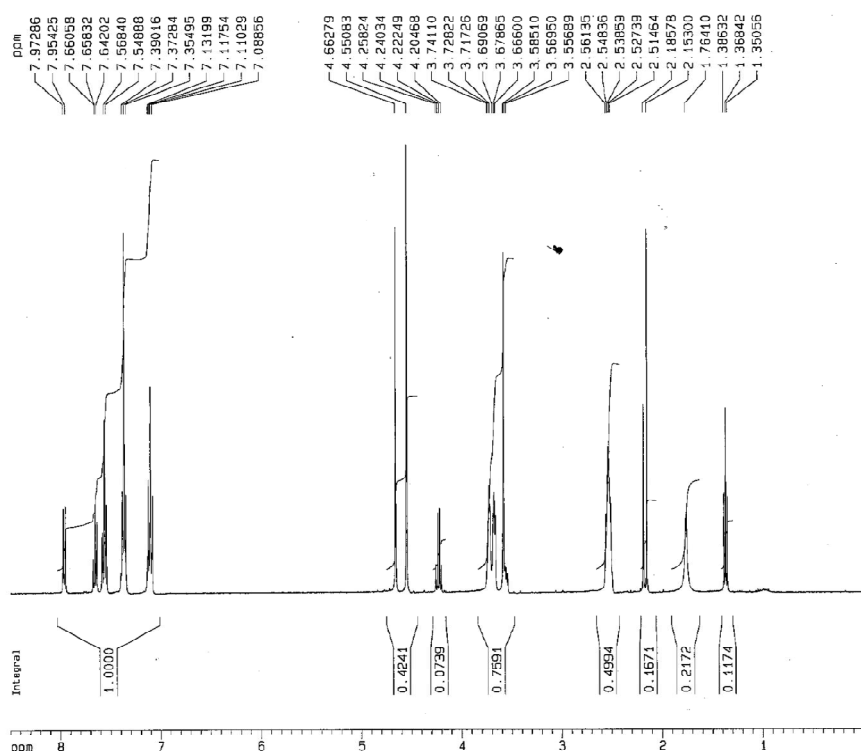
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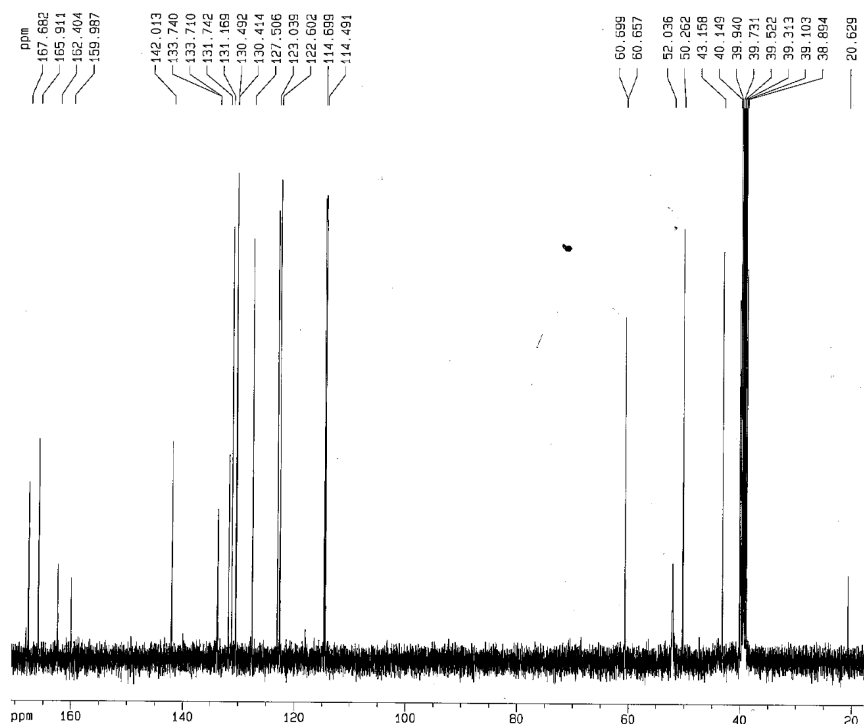
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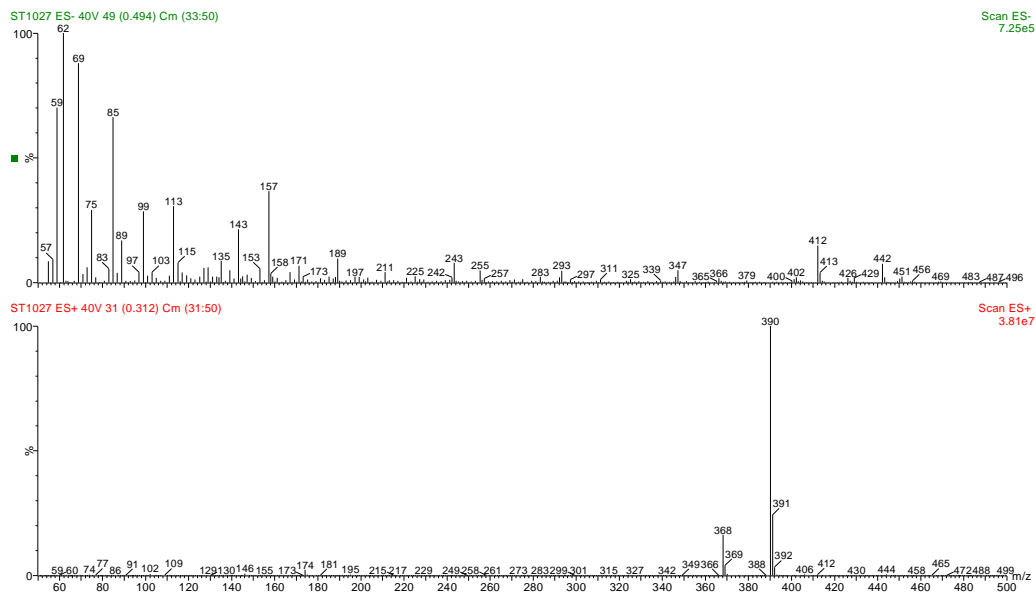
Email maxine.roberts@ansto.gov.au

Characterisation of 9

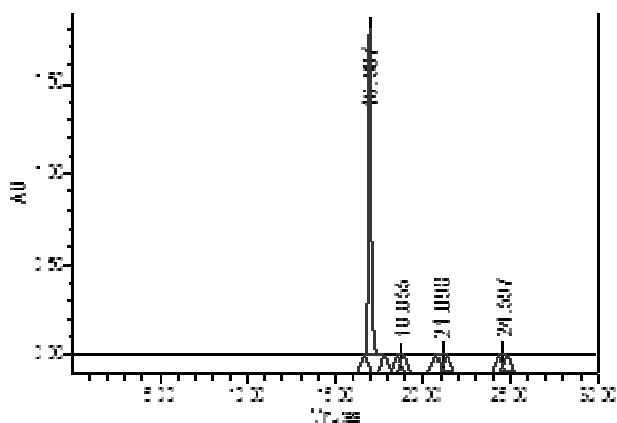
Supplementary 1.1: ¹H NMR spectrum of **9** in CDCl₃.



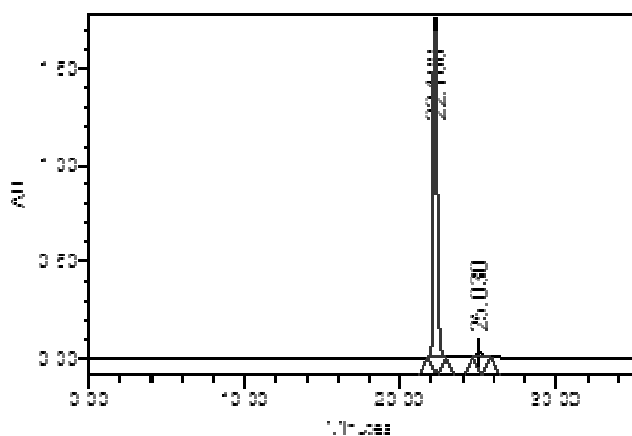
Supplementary 1.2: ^{13}C NMR spectrum of **9** in d_6 -DMSO.



Supplementary 1.3: LRMS spectrum of **9**. Mass Spectroscopy direct infusion result indicated the presence of intact product at ES⁺ m/z 368 $[\text{M}+\text{H}]^+$, 390 $[\text{M}+\text{Na}]^+$.



Supplementary 1.4: HPLC QC chromatograph of **9**. Mobile phase ACN/H₂O/100mM pH8 Ammonium Bicarbonate; purity: 99.45%; retention: 16.98 mins. Purity analysis was obtained by integration of the area under the curve.



Supplementary 1.5: HPLC QC chromatograph of **9**. Mobile phase MeOH/H₂O/100mM pH8 Ammonium Bicarbonate Purity: 97.41%; retention: 22.18 mins. Purity analysis was obtained by integration of the area under the curve.

Equipment used for HPLC QC: Waters 600 controlled Model 60F; Column used: AlltimaC18 150 x 4.6 mm; UV: Waters PDA, model 299, 254 nm; Injection amount: 5 μ L in CH₃OH; solvent condition: grad 1 mL/min, A) ACN or MeOH (10-90%), B) H₂O (80-0%), D) 100 mM pH8 ammonium bicarbonate (10%) over 20 mins.

Elemental Composition

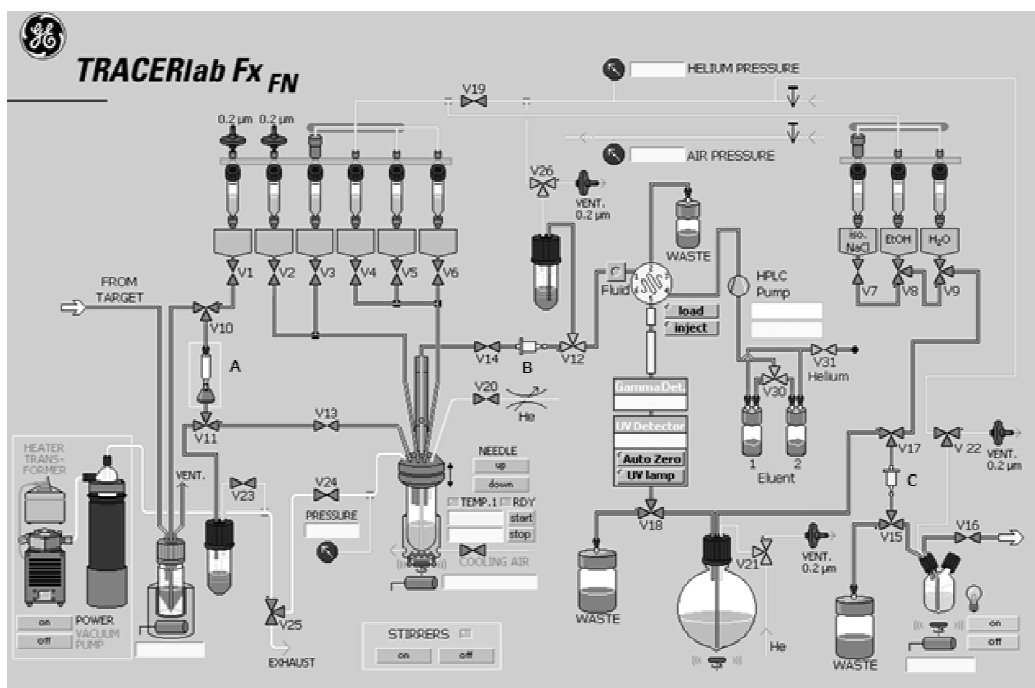
Date : 24-JUL-2008

File:SM1027 Ident:13_26 SMO(1,3) BSOB(128,15,-3.0)
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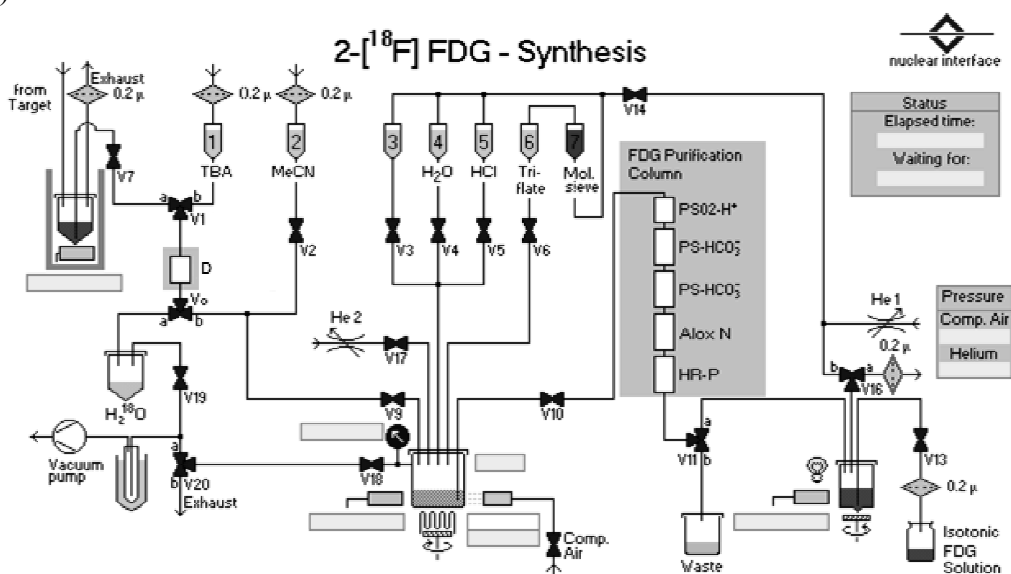
Mass	PM	mDa	Calc. Mass	DEE	C	H	N	O	F
367.169849	10.0			-0.5 20.0	0 200	0 400	0 9	0 5	0 1
367.169849	-0.1	0.0	367.169805	15.5	26	23		2	
	-0.7	-0.2	367.169605	12.0	21	22	3	2	1
	3.0	1.1	367.170948	11.5	23	24		3	1
	-3.8	-1.4	367.168463	16.0	24	21	3	1	
	-4.3	-1.6	367.168263	12.5	19	20	6	1	1
	4.9	1.8	367.171650	8.0	13	21	9	4	
	-7.4	-2.7	367.167120	16.5	22	19	6		
	-8.0	-2.9	367.166925	7.5	18	24	2	5	1
	-8.0	-2.9	367.166920	13.0	17	18	9		1
	8.0	2.9	367.172793	4.0	10	22	9	5	1
	8.6	3.1	367.172993	7.5	15	23	6	5	
	10.3	3.8	367.173628	16.0	26	22	1		1
	-11.1	-4.1	367.165782	11.5	21	23	2	4	
	-11.6	-4.3	367.165583	8.0	16	22	5	4	1
	-14.7	-5.4	367.164440	12.0	19	21	5	3	
	-15.3	-5.6	367.164240	8.5	14	20	8	3	1
	15.9	5.8	367.175673	12.0	18	21	7	2	
	-18.4	-6.8	367.163097	12.5	17	19	8	2	
	19.0	7.0	367.176816	8.0	15	22	7	3	1
	19.5	7.2	367.177016	11.5	20	23	4	3	
	22.6	8.3	367.178159	7.5	17	24	4	4	1
	23.2	8.5	367.178359	11.0	22	25	1	4	
	-24.0	-8.8	367.161052	16.5	25	20	2		1
	-25.7	-9.4	367.160417	8.0	14	21	7	5	
	26.3	9.6	367.179496	12.5	18	20	8		1
	26.3	9.7	367.179501	7.0	19	26	1	5	1
	26.8	9.8	367.179696	16.0	23	21	5		

Supplementary 1.6: Elemental composition of 9.

a)



b)



Supplementary 1.7: Schematic diagram of a) GE TRACERlab™ FX_{FN} automated synthesis module used to synthesise **11**; b) in-house modified Nuclear Interface [¹⁸F]FDG automated synthesis module used to synthesise [¹⁸F]**9**.

GE TRACERlab FX _{FN} Reactor		Nuclear Interface [¹⁸ F]FDG synthesis module	
Position	Reagents/material	Position	Reagents/material
V1	K ₂ CO ₃ (2 mg), Kryptofix 2.2.2 (10 mg) in CH ₃ CN/H ₂ O (1 mL, 80:20)	V1	Methanol (1 mL)
V2	Precursor 10 (2 mg) in DMF (1 mL)	V3	Precursor 8 (4.5 mg) in CH ₃ OH (0.4 mL) and AcOH (20 μL)
V3	HCl (0.1 M aq, 12 mL)	V4	NaCNBH ₃ (2 mg) in CH ₃ OH (0.4 mL)
V4	H ₂ O (5 mL)	V5	H ₂ O (2 mL)
A	Sep-Pak Light QMA	D	Sep-Pak Plus C18

Supplementary 1.8: Preparation of reagents for the two-step, two-pot automated radiosynthesis of [¹⁸F]**9** using the GE Tracerlab FX_{FN} and Nuclear Interface [¹⁸F]FDG synthesis module.