Radical cyclisation studies of β-nitroamines from the nitro-Mannich reaction

Abil Aliev, James C. Anderson,* Mérina Corpinot and Emily S. J. Gascoigne

Supplementary Information

General experimental.

Structural elucidation of **31** and data correlation table.

Data correlation tables for compounds **34a**, **35a**, **37**, **39**, **40a** major, **40a** minor, **40b** major, **40b** minor.

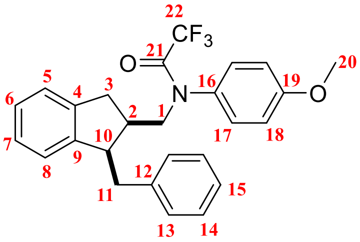
X-ray crystallography data for compounds **32a** and **35a**

General Experimental. All anhydrous chemistry was performed using a Schlenck apparatus and standard syringe techniques; all glassware was flame-dried and reactions were conducted under an inert atmosphere (argon or nitrogen). Room temperature (rt) denotes a value of 20 – 25 °C, and any temperatures stated reflect the temperature of the external heating/cooling environment. Reaction cooling to 0 °C was performed in an ice-water bath, to – 20 °C in an ice-salt bath, to – 40 °C with a dry ice/acetonitrile bath, and temperatures of – 78 °C were obtained usually by a dry ice/acetone bath, or a liquid nitrogen/EtOAc bath. Thin layer chromatography was carried out using Polygram® SIL G/UV254 0.25 mm silica backed TLC plates, with visualisation by UV light (254 nm) and $KMnO_4$ or anisaldehyde dip. Flash column chromatography was performed using Gedran® silica gel 60, 40 – 63 µm; the solvent system used is stated in each case. Removal of solvents *in vacuo* was performed using Büchi rotary evaporators with the house vacuum, diaphragm or Vacuubrand vacuums.

All ^1H, ^{13}C and ^{19}F NMR data were obtained using Bruker AMX 300 MHz, Bruker AVANCE III 400 MHz, Bruker AVANCE 500 MHz or Bruker AVANCE III 600 MHz machines, at room temperature unless stated. Data was manipulated directly using TopSpin (3.2). Unless otherwise stated, NMR data was obtained as a dilute solution in $CDCl_3$, chemical shifts (δ) are reported in parts per million (ppm) relative to the solvent peak values of δ = 7.26 ppm (^1H NMR) and 77.1 ppm (^{13}C NMR) for $CDCl_3$. Multiplicities are reported as s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, m = multiplet, br refers to a broad signal, apt = apparent. Coupling constants (*J*) are measured in Hertz (Hz). Where appropriate, additional NMR techniques have been used to aid assignment (COSY, DEPT, HSQC, HMBC and NOE experiments). Mass spectroscopy analysis data were collected using Thermo Finnigan Mat900xp (EI/CI) or Waters LCT premier XE (ESI) machines. Infrared data were collected on a Shimadzu 8700 FT-IR machine as a neat sample (thin film). Elemental analysis was carried out on an Exeter Analytical Inc. EA440 horizontal load analyser. Melting points are reported uncorrected, and were recorded on a Griffin melting point apparatus.

Purification of laboratory reagents. With the exception of specifically mentioned examples, all commercial reagents and solvents detailed were used as supplied or purified by the standard techniques described.[269] The concentration of commercially-sourced organolithium reagents was verified by titration with *N*-benzylbenzamide at – 40 °C.[270] Dry dichloromethane (CH_2Cl_2), diethyl ether (Et_2O), toluene, tetrahydrofuran (THF) or hexanes were obtained from a solvent tower, in which degassed solvents were passed through two columns of activated alumina and a 7 micron filter under 4 bar pressure. Purification and drying of alcohols, aldehydes, amines or alkenes was carried out via standard procedures, and the resultant purified substances stored under argon at the correct temperature.[269] Benzene used for radical reactions was purified to remove traces of thiophene as described.[269] Recrystallisation of AIBN (2,2'-azobis(2-methylproionitrile)) was carried out from Et_2O, the reagent was stored under argon between – 5 and – 20 °C. Diiodoethane was washed with sodium thiosulfate solution, dried and stored under argon at – 20 °C. Activation of 3 or 4 Å molecular sieves was performed via heating under high vacuum.

Structural elucidation of 31.



31

Analysis by ^1H and ^{13}C NMR techniques confirmed that this was an indane derivative (Full correlation table is given in the appendix). Initial analysis of the molecule was based around the assignment of the CH_2N peaks at δ 3.74 ppm (dd, *J* = 13.3, 5.3) and δ 4.31 ppm (dd, *J* = 13.3, 9.8), which corresponded to a CH_2 unit at δ 52.0 ppm by HSQC analysis. Such deshielded peaks for a CH_2 unit could be assigned to an electron-poor amine, in this case adjacent to the 2,2,2-trifluoroacetamide (# = 1); the shift at δ 52.0 ppm was low in comparison with the *C*HN peak for the initial denitration non-cyclised product **2** (Scheme 1, main paper) (δ 59.9 ppm), due to the loss of the adjacent aromatic ring. Retention of the PMP and TFA groups were confirmed by ^{13}C NMR, with the usual quaternary carbons (δ 131.2 ppm, Ar*C*N) and (δ 159.9 ppm, Ar*C*O) and methyl peak (δ 55.6

ppm, OCH_3, also δ 3.84 ppm (3H, s, OCH_3) by ^{1}H NMR) and ^{19}F NMR, with the expected peak at δ –66.9 ppm (3F, s, CF_3). It was clear that no aromatic ring had been lost, due to the presence of 9 Ar*H* by ^{1}H NMR, hence it was hypothesised that a rearrangement had occurred, with a proposed structure **31**. The proposed structure was investigated by ^{1}H and ^{13}C NMR experiments, including HSQC and HMBC; a NOESY experiment was run to give an idea of the likely stereochemistry across the indane ring. It was known by DEPT and HSQC analysis that 3 CH_2 units were present in the molecule, at δ 35.2, 35.4 and 52.0 ppm by ^{13}C NMR. Ruling out the CH_2N peak at δ 52.0 ppm, carbons 3 and 11 were assigned as either δ 35.2 or 35.4 ppm. The remaining aliphatic *C*H peaks at δ 41.5 and 48.0 ppm could be speculatively assigned as carbons 2 and 10 respectively due to their difference in shifts. Carbon 10 (δ 48.0 ppm) is adjacent to the aromatic ring of the indane and would likely be shifted further downfield than carbon 2. With a starting point of the CH_2N (# = 1) peaks as previously mentioned, the coupling of these protons and the attached carbon to neighbouring protons/carbons was investigated (see correlation table below). It was found that carbon 1 coupled to protons at positions 2 (δ 2.72 ppm, m) and 3 (δ 2.93 ppm, 2H, d, *J* = 8.2), confirming them in close proximity. Protons 2 and 3 were coupled to signals at δ 41.5 and 35.2 ppm in the HSQC spectrum; carbon 10 coupled by HSQC to a proton at δ 3.36 ppm (1H, dt, *J* = 10.2, 6.5). Carbon 10 also coupled to protons at 1, 1’, 2, 3, and protons 11 and 11’, which were assigned by this correlation, their benzylic shift (by ^{1}H and ^{13}C NMR of the corresponding carbon at δ 35.4 ppm), and HMBC couplings of C-11 to aromatic protons. Carbon 11 and its corresponding protons 11 and 11’ by HSQC were further confirmed by couplings of C-11 to protons 2 and 10; a lack of coupling to protons 1,1’ suggested that carbon 11 was the benzylic peak *exo* to the ring, and not in fact position 3. Carbon 3 is coupled to 1, 1’ 5 and 10 which confirms its closer proximity to CH_2N protons 1,1’ and its presence in the indane ring. Further COSY analysis confirms the arrangement of the atoms with couplings between 1-2, 1’-2, 2-3, 2-10, 10-11, and 10-11’ as the most diagnostic peaks.

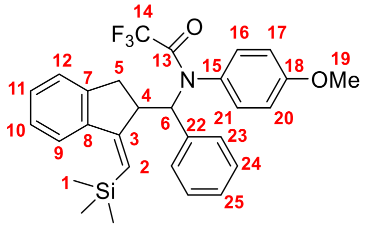
δ_H / ppm	Multipl.	J_{HH} / Hz	#	COSY	δ_C / ppm	*HMBCs*	*NOEs*
2.61	dd	13.3, 10.3	**11’**	10, 11	35.4	-	
2.72	m	-	**2**	1, 1’, 3	41.5	1,1’,10,3,11,11’	**10** (s)
2.90	dd	13.4, 6.0	**11**	10, 11’	35.4	13,2,10	
2.93	2H, d	8.2	**3**	2	35.2	5,1,1’,(10	
3.36	dt	10.2, 6.5	**10**	2, 11, 11’	48.0	8,1,1’,3,11,11’,2	**2** (s)

3.74	dd	13.3, 5.3	**1’**	1, 2	52.0	-
3.84	s		**20**		55.6	-
4.31	dd	13.3, 9.8	**1**	1’, 2	52.0	3,2
6.42	d	7.5	**8**		125.0	6
6.87 – 6.92	m		**18**		~115.0 br	
6.94	td	7.5, 1.0	**7**		125.8	5
6.96 – 7.00	m		**13**		129.4	3,3’
7.13	td	7.5, 0.9	**6**		126.9	8
7.15	m		**17**		~130 br	
-	m		**15**		126.2	13o
7.25	d	7.5	**5**		124.7	3
	m		**14**		128.3	
-		$^1J_{CF}$= 288.6	**22**		116.6	
-			**16**		131.2	17,18,1,1’
-			**12**		139.8	14,10,11,11’
-			**4**		141.8	6,8,10,3
-			**9**		145.6	5, 7, 10,3,11,11’
-		$^2J_{CF}$= 34.9	**21**		157.5	1, 1’
-			**19**		159.9	17, 18, 20

Structural elucidation of 32a and 32a’.

Full characterisation of both compounds was undertaken, with confirmation of the molecular ion peaks at 498 (M + H$^+$) and 497 (M$^+$) by MS analysis of **32a** and **32a’**. The indane ring in both products was confirmed as for **31**, using ^{1}H NMR and ^{13}C techniques, HSQC and HMBC providing the structural information necessary to assign relative stereochemistry.

Data correlation table for 34a.

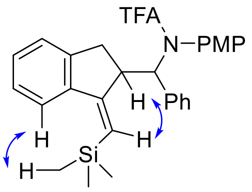


34a

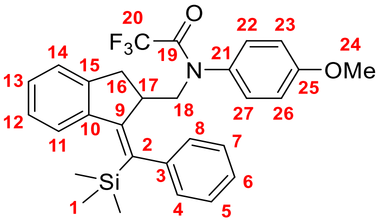
δ_H / ppm	**Mult.**	**J_{HH} / Hz**	**#**	**COSY**	**δ_C / ppm**	***HMBCs***	***NOESY***
0.29	9H, s		**1**		0.05		9
2.27	1H, apt d	16.7	**5**	4	35.5	3, 4, 6, 7, 8, 12	
3.04	1H, dd	16.6, 7.5	**5’**	4, 12	35.5	4, 6, 7, 8	
3.78	3H, s		**19**		55.5	18	
4.21	1H, br dd	10.8, 7.5	**4**	5’, 6	48.0	5, 5’, 6, 7	2
4.55	1H, br d	10.8	**6**	4	71.1 (br)	4, 5, 5’	
5.99	1H, s		**2**	4	124.8	1, 2, 4, 6, 8	4, PMP
6.67	1H, dd	8.9, 3.0	**PMP**		114.3		
6.71 – 6.74	1H, m		**PMP**		131.2		2
6.80	1H, dd	8.7, 2.7	**PMP**		113.5		
7.07	1H, m		**PMP**		130.1		2
7.11	1H, d	7.5	**12**	5’	125.7	5, 8	
7.18 -7.31	7H, m		**ArH**				

7.67	1H, d	7.7	**9**	124.6	2, 7	1
			15	130.4		
			22	137.3		
			8	140.4	2,5,5’,12	
			7	145.7	4,5,5’,9	
		35.5	**14**	156.9		
			3	159.3	2, 5, 9	
			18	159.6	19	

Key NOESY interactions to confirm geometry of alkene.



Data correlation table for 35a.

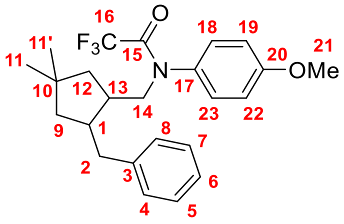


35a

δ$_H$ / ppm	Mult.	J_{HH} / Hz	#	δ$_C$ / ppm	*HMBCs*
0.07	9H, s		**1**	0.5	1
2.72	1H, apt d	15.4	**16**	33.5	13, 14, 15, 17, 18
2.90	1H, dd	13.3, 3.3	**18**	52.3	16, 17
2.93 – 3.03	2H, m		**16 & 17**	33.5 & 42.1	**16** (12, 13, 15, 17, 18) **17** (qs)
3.85	3H, s		**24**	55.6	(qs)
4.01	1H, dd	13.1, 11.5	**18**	52.3	16, 17,

6.60 – 7.17	9H, br m			
7.24	1H, apt t	**12**	126.0	
7.28	1H, dd	**13**	128.8	
7.32	1, d	**14**	126.3	15
7.66	1H, d	**11**	125.5	15
		21	130.0	18
		2	139.4	1, 17
		15	139.8	11, 14, 16,
		3	145.2	(Ar)
		10	146.6	11, 16, 17,
		9	153.5	11, 16, 17, 18
		19	157.6	18
		25	159.3	24

Data correlation table for 37.

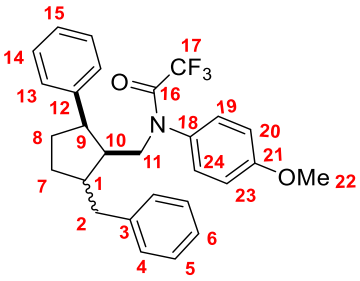


37

δ$_H$ / ppm	Mult.	J_{HH} / Hz	#	COSY	δ$_C$ / ppm
0.92	3H, s	-	**11**		30.6
1.11	3H, s	-	**11'**		31.4
1.34	1H, dd	13.0, 8.5	**9**		46.3
1.40	1H, dd	13.0, 6.6	**9**		46.3
1.50	1H, dd	13.0, 7.8	**12**		44.7
1.58	1H, dd	13.0, 7.3	**12**		44.7

2.27	1H, m		**13**	12, 14	38.8
2.38	1H, m		**1**	9, 13	42.4
2.43	1H, dd	12.8, 10.7	**2**	4/8	36.5
2.70	1H, dd	12.8, 4.7	**2**	4/8	36.5
3.49	1H, dd	13.1, 4.9	**14**	13	52.4
3.85	3H, s	-	**21**		55.6
4.25	1H, dd	13.0, 11.0	**14**		52.4
6.91	2H, apt d	8.7	**19 & 22**		114.0, 114.7
7.08	2H, apt d	7.8	**4 & 8**		128.8
7.11	2H, d	8.6	**18 & 23**		129.4, 130.0
7.16	1H, tm	7.4	**6**		125.9
7.22 – 7.25	2H, m	**-**	**5 & 7**		128.4
			10		37.4

Data correlation table for 39.

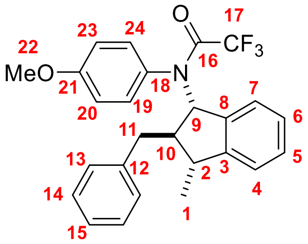


39

δ_H / ppm	Mult.	J_{HH} / Hz	#	COSY	δ_C / ppm	*HMBCs*	*NOESY*
1.54	m	-	**7**	7’, 1/2	30.9		
1.61	1H, m	-	**8**	8’	34.8		
1.74	1H, m	-	**7’**	7, 8’, 1	30.9		
2.25 – 2.33	2H, m	-	**8’ &**	(7, 8’, 7’)	34.8		

			10	(11, 8, 9)	47.6		**1** (w) **9** (s)
2.40 – 2.47	2H, m	-	**2 &**	7	35.7,		
			1		44.1		**9** (w) **10** (w)
2.77	1H, m	-	**2’**	1/2	35.7		
3.13	1H, apt q	8.4	**9**	10	49.1		**10** (s) **1** (w)
3.62	1H, dd	13.5, 6.0	**11**	10	52.7		
3.79	3H, s	-	**22**				
4.28	1H, dd	13.4, 9.1	**11**	10	52.7		
			18		131.0 (q)	11	
			3		141.2 (q)	2	
			12		146.6 (q)	9	
			16		157.6 (q)	11	
			21		159.5 (q)	22	

Data correlation table for 40a major.

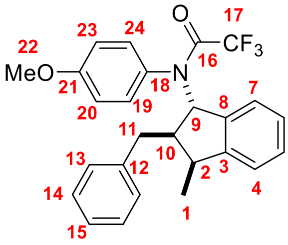


40a major

δ_H / ppm	**Mult.**	***J_{HH}* / Hz**	**#**	**COSY**	**δ_C / ppm**	***HMBCs***	***NOESY***
0.97	3H, d	7.0	**1**	2, 10(w)	18.8	2, 9(w), 10	
1.99	tdd	8.7, 7.6, 6.1	**10**	1(w), 2, 9, 11’	51.4	1, 2, 11, 11’, 9	**2** (-) **11** (m)

2.85	m	-	**2**	1, 10	42.6	1, 10,11, 11’, & ArH	
2.95	dd	13.7, 6.0	**11**	10, 11’	39.2	2, 9, 10, ArH	
3.03	dd	13.7, 7.5	**11’**	10, 11	39.2	2, 9, 10, ArH	
3.76	3H, s		**22**		55.4	-	
6.28	d	8.8	**9**	2, 10	67.0	10, 11, 11’	**2** (w) **10** (m) **11** (m)
6.37	Apt d	8.6	**PMP**		113.6 –	-	
6.59	dd	8.9, 2.9	**PMP**		132.1		
6.61-6.65	m	-	**PMP**				
6.76	dd	8.8, 2.9	**PMP**				
7.09-7.13	2H, m	-	**ArH**				
7.23-7.39	7H, m	-	**ArH**				
		$^1J_{CF}$= 288.3	**17**		116.6	-	
			18		127.9	9, PMP	
			8		139.6	2, 9, 10	
			12		139.6	10, 11, 11’	
			3		146.7	1, 2, 9, ArH	
		$^2J_{CF}$= 35.1	**16**		158.2		
			21		159.8		

Data correlation table for 40a minor.



40a minor

δ_H / ppm	*Mult.*	*J_{HH} / Hz*	*#*	*COSY*	*δ_C / ppm*	*HMBCs*	*NOESY*
1.12	3H, d	7.3	**1**	(2/11')[a] 9 (w)	17.3	(2/11')[a], 10	
2.60	tdd	9.2, 7.9, 6.7	**10**	(2/11')[a], 9	39.3	1, 11	**2** (s) **9** (w) **11** (-)
2.90	dd	14.5, 9.2	**11**	10	34.4	1, (2/11')[a], 9, 10	
2.96-3.04	2H, m		**11' & 2**	1, 10	34.4 & 45.3	1, (2/11')[a], 9, 11	
3.77	3H, s		**22**	-	55.5		
6.24	d	9.2	**9**	10, ArH	66.3	(2/11')[a], 10, 11, ArH	**10** (w) **2** (-) **11** (m)
6.61-6.65	m		**PMP**		113.6 - 132.1		
6.70	dm	8.7	**PMP**				

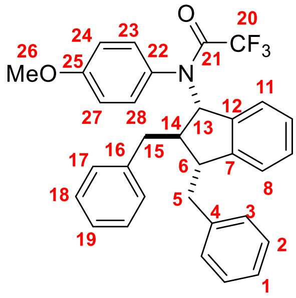
6.83	m		**PMP**		
7.18	2H, apt d	7.5	**ArH**		
7.23-7.39	7H, m		**ArH**		
		$^1J_{CF}$= 289.2	**17**	116.6	
			18	127.8	9, PMP
			8	139.1	9, ArH
			12	140.0	10, 11, 11’, ArH
			3	148.7	1, 2/11’, 9, ArH
		$^2J_{CF}$=34.6	**16**	158.5	9
			21	160.0	PMP

[a]Coincident signals

Structural elucidation of diastereoisomers 40b

Assignment of the major and minor diastereoisomers was conducted by ^{1}H NMR, using $^3J_{HH}$ coupling values for the indane ring protons and NOESY interactions. Analysis of *J* couplings showed that for the major diastereoisomer $^3J_{1,2}$ = 7.1 Hz, $^3J_{2,3}$ = ~7 Hz, suggesting a common orientation of protons H_1-H_2 to H_2-H_3 in the *cis*-region as previously verified for **32a**. However NOESY correlations showed H_1-H_2 (w), $H_{2'}$-H_3 (w) and H_1-H_3 (m), suggesting a *trans,trans*-configuration. This diastereoisomer demonstrates the difficulty in assignment of indanes, and the requirement for multi-faceted evidence for assignment. In comparison the minor isomer was tentatively assigned as the *trans,cis*-isomer, with a larger coupling constant for $^3J_{1,2}$ = 9.2 Hz, which more convincingly suggested a *trans*-orientation of these protons, further corroborated by a weak NOESY interaction. The orientation of protons H_2-H_3 was assigned as *cis*- due to the strong NOESY interaction between them, a coupling constant $^3J_{2,3}$ = 7.5 Hz and also a lack of interaction between protons H_1-H_3. The C*H*N ^{1}H NMR shifts of both protons H_1 in **40b** were also very similar to each other (δ 6.31 ppm major and 6.28 ppm minor) and to those of **40a** (δ 6.28 ppm and 6.24 ppm) suggesting little change in environment between H_1-H_2 in all four molecules (*trans*-).

Data correlation table for 40b major.

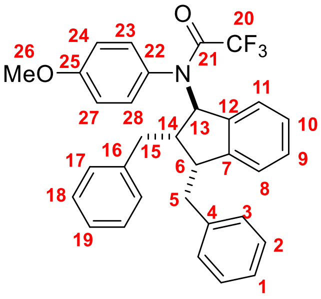


40b major

δ$_H$ / ppm	*Mult.*	*J$_{HH}$ / Hz*	*#*	*δ$_C$ / ppm*	*HMBCs*	*NOESY*
2.21	1H, dtd	9.6, 7.1, 4.8,	**14**	47.5		13 (w)
2.24	1H,dd	13.4, 8.2	**5**	41.2		
2.51	1H, dd	13.3, 4.6	**15**	41.0		
2.70	1H, dd	13.4, 9.6	**15’**	41.0		
2.79	1H, dd	13.6, 6.3	**5’**	41.2		
3.13 – 3.18	m		**6**	49.8		13 (m) 14 (w)
3.78	3H, s	**-**	**26**	55.5		
6.17	1h, d	7.8	**PMP**			
6.31	1H, d	7.1	**13**	67.3		
6.42	1H, dd	8.7, 2.6	**PMP**			
6.54	1H, dd	8.9, 3.0	**PMP**			
6.75	1H, dd	8.8, 3.0	**PMP**			
6.91	2H, m	-	**Ar**			
7.05	3H, apt d	7.0	**Ar**		12	
7.15 – 7.35	8H, m	-	**Ar**		12, 7	
7.37	1H, d	7.4	**Ar**		7	
		288.7	**20**	116.6		
			22	128.0	PMP	

	4	139.3	5
	16	139.6	15
	12	140.2	13
	7	145.6	5, 6, 13
34.8	**21**	157.6	
	25	159.8	

Data correlation table for 40b minor.

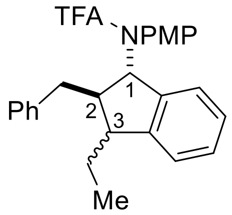


40b minor

δ_H / ppm	Mult.	J_{HH} / Hz	#	COSY	δ_C / ppm	HMBCs	NOESY
2.44	1H, dd	13.4, 8.2	**5**		36.5	6, 14	13 (m)

2.69	1H, m		**14**	6, 13, 15	46.0	5’, 6, 13, 15	6 (s)
3.05	2H, m		**15**	14	34.1	13, 14	13 (s)
3.10	1H, ddd	10.8, 7.5, 5.0	**6**	5, 14	46.2	5, 14	
3.13 – 3.18	1H, m		**5’**		36.5	6, 14	
3.78	3H, s		**26**		55.5		
6.24	1H, d	7.3	**Ar**				
6.28	1H, d	9.2	**13**	14	66.0	6, 14, 15	5 (m) 5’ (s)
6.63	1H, dd	8.9, 2.9	**PMP**				
6.69	1H, dd	8.9, 2.5	**PMP**				
6.78	2H, m		**Ar**				
6.86	1H, dd	8.8, 2.9	**PMP**				
6.95	1H, m		**PMP**				
7.00	1H, apt t	7.4	**Ar**				
7.15 – 7.35	9H, m		**Ar**				
7.40	1H, d	7.6	**Ar**				
		289.4	**20**		116.7		
			22		127.8		
			16		139.5		
			12		139.6		
			4		139.8		
			7		145.7		
		34.7	**21**		158.6		
			25		160.1		

Structural elucidation of major diastereoisomer of 40d

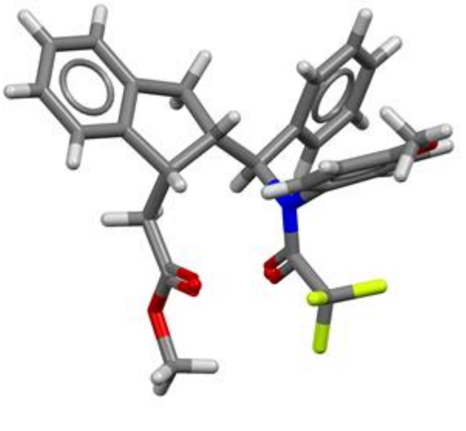


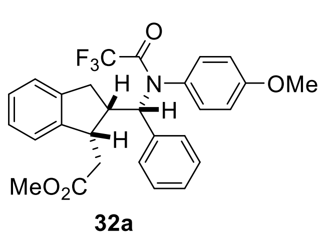
40d 12%
dr 70:30
trans, trans: *trans, cis*

Analysis of the stereochemistry by NOESY interactions gave correlation between protons H_1-H_2 (w), H_2-H_3 (w) and H_1-H_3 (s) suggesting a *trans,trans*-orientation. Analysis of $^{3}J_{HH}$ small couplings $^{3}J_{H1H2} = 7.6$, $^{3}J_{H2H3} = \sim 7.1$ Hz, in the range of observed values for *cis*-dialkyl substituted indanes. However the similar values across H_1-H_2, H_2-H_3 suggest a *trans,trans*-environment due to the similar $^{3}J_{HH}$ values suggesting a similar dihedral angle across both bonds. The chemical shift of the C*H*N proton (H_1, δ 6.30 ppm) is also close to those of **40a** (δ 6.28 and 6.24 ppm) and **40b** (δ 6.31 and 6.28 ppm) suggesting a similar *trans*-H_1H_2 environment. The relative stereochemistry was therefore tentatively assigned as *trans,trans*- due to the consideration of all three pieces of data.

X-ray crystallography data. The single crystal X-ray diffractometer is equipped with an Atlas CCD Detector. All data sets collected at 150 K using CuKα radiation ($\lambda = 1.54184$ Å). The data were acquired and processed using the CrysAlis^{Pro} program.[298] The datasets were corrected for Lorentz and polarization effects. Structure solution and refinement were accomplished using SHELXS-97 and SHELXL-97,[299] respectively. The crystal structures were solved using direct methods. All non-hydrogen atoms were refined anisotropically, while hydrogen atoms associated with carbon and oxygen atoms were refined isotropically in geometrically constrained positions. Hydrogen atoms affiliated with nitrogen atoms were refined isotropically in positions identified in the difference Fourier map. Crystallographic and refinement parameters for crystal structures in section 4.4.2.

methyl 2-((1*R**,2*R**)-2-((*R**)-phenyl(2,2,2-trifluoro-N-(4-methoxyphenyl)acetamido)methyl)-2,3-dihydro-1H-inden-1-yl)acetate (32a)

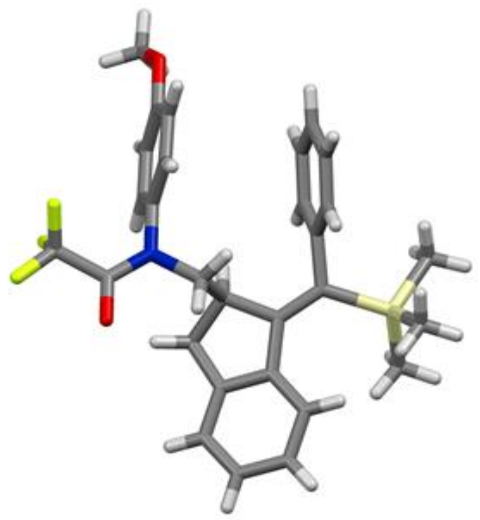


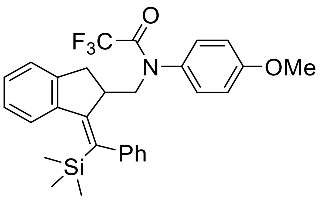


32a

chemical formula	$C_{28}H_{26}F_3NO_4$	
M_r/g mol^{-1}	497.50	
crystal system	monoclinic	
space group	$P\ 2_1/c$	
Unit cell dimensions	a = 15.1930(3) Å	α = 90°
	b = 13.0292(2) Å	β = 110.560(3)°
	c = 12.8223(3) Å	γ = 90°
Volume	2376.54(9) Å^3	
Z	4	
Density (calculated)	1.39 cm^{-3}	
F(000)	1040	
μ(CuK_α)	0.912 mm^{-1}	
T/K	150(2)	
	−18 → 18	
index range	−15 → 15	
	−15 → 10	
collected reflections	34570	
unique reflections	4198	
R_{int}	0.1108	
reflections with $I > 2\sigma(I)$	3452	
no. parameters	325	
$R(F)$, $F > 2\sigma(F)$	0.0426	
$wR(F^2)$, $F > 2\sigma(F)$	0.11	
$R(F)$, all data	0.0529	
$wR(F^2)$, all data	0.1171	
Δ_r (min., max.) e Å^{-3}	−0.225, 0.238	

(*Z*)-2,2,2-trifluoro-*N*-(4-methoxyphenyl)-*N*-((1-(phenyl(trimethylsilyl)methylene)-2,3-dihydro-1*H*-inden-2-yl)methyl)acetamide (35a)





35a

chemical formula	$C_{29}H_{30}F_3NO_2Si$
M_r/g mol^{-1}	509.64

crystal system	triclinic	
space group	*P -1*	
Unit cell dimensions	a = 10.1262(3) Å	α = 115.009(4)°
	b = 12.1318(4) Å	β = 96.041(3)°
	c = 12.5425(4) Å	γ = 104.045(3)°
Volume	1316.22(8) Å^3	
Z	2	
Density (calculated)	1.226 cm^{-3}	
F(000)	511	
μ(CuK_α)	1.162 mm^{-1}	
T/K	150(2)	
index range	−12 → 12	
	−13 → 14	
	−14 → 14	
collected reflections	18582	
unique reflections	4623	
R_{int}	0.0474	
reflections with $I > 2\sigma(I)$	3884	
no. parameters	329	
$R(F)$, $F > 2\sigma(F)$	0.0638	
$wR(F^2)$, $F > 2\sigma(F)$	0.1751	
$R(F)$, all data	0.0814	
$wR(F^2)$, all data	0.1853	
Δ_r (min., max.) e Å^{-3}	-0.378, 1.622	