Imprinted polymers for chiral resolution of (+/-)-ephedrine, 2: Probing pre-polymerisation equilibria in different solvents by NMR.

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Supplementary Information

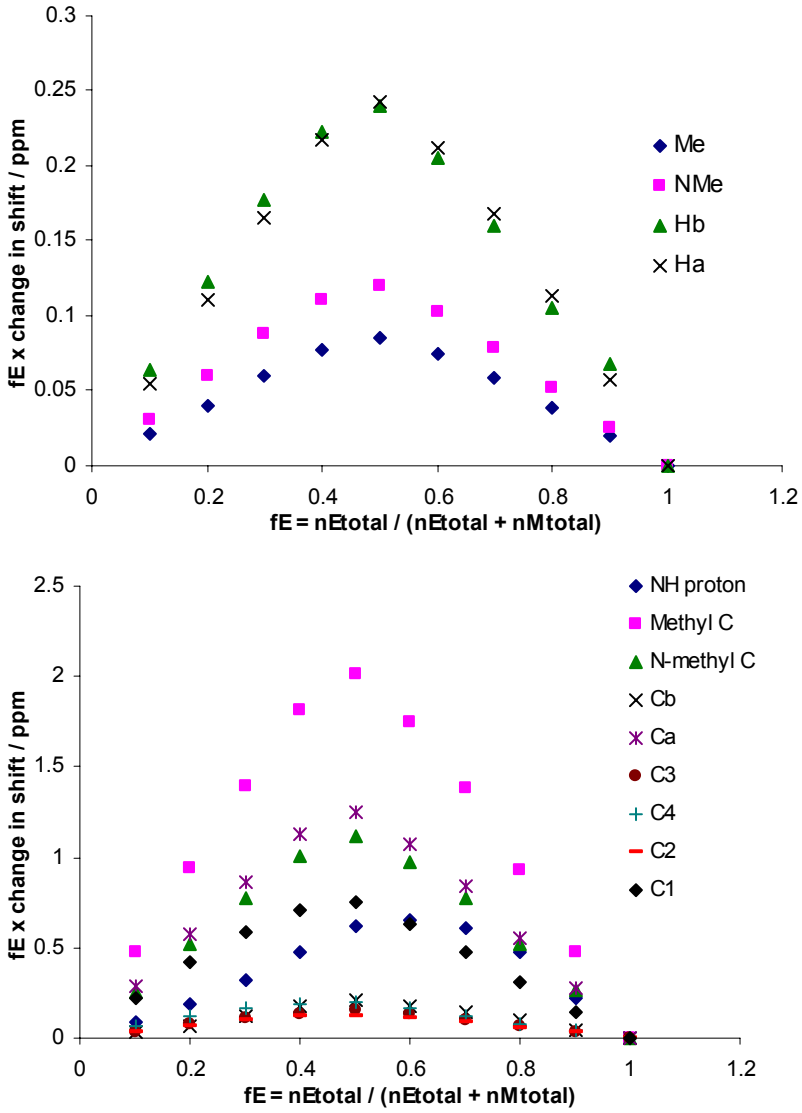
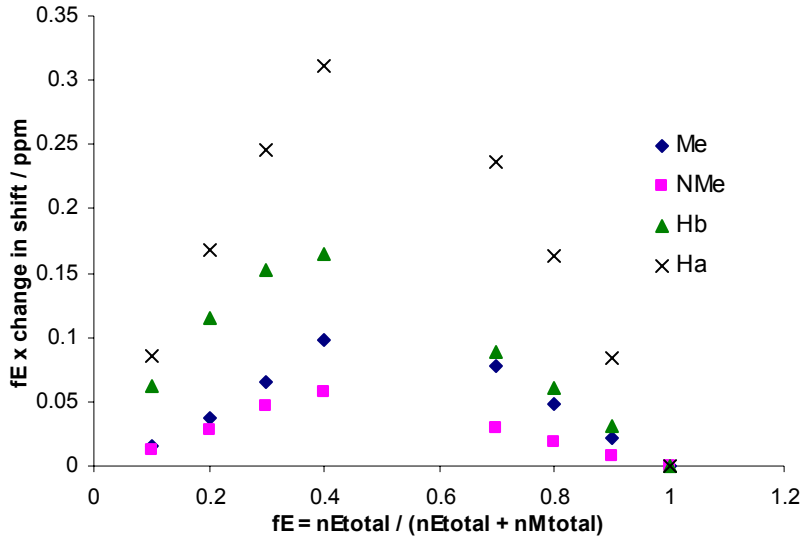


Figure S1 Job plots for the association between (-)-ephedrine and MAA in acetonitrile-d_3. a) non-exchangeable protons b) NH protons and carbon nuclei.



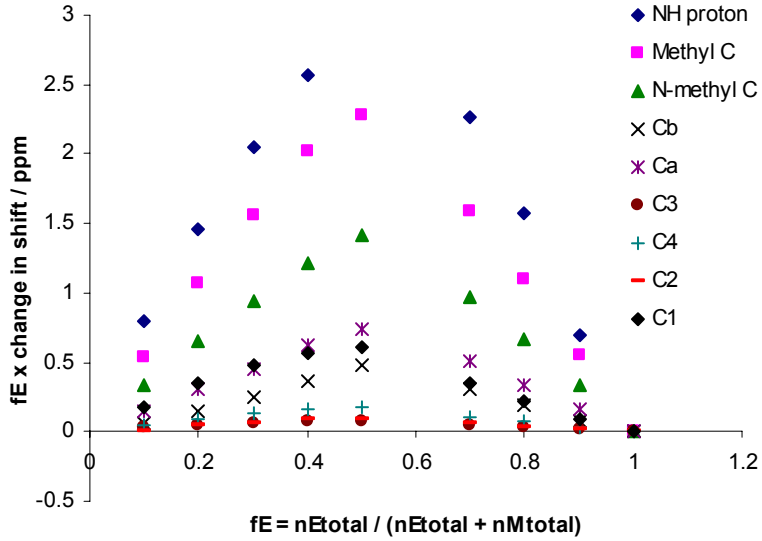


Figure S2 Job plots for the association between (-)-ephedrine and MAA in toluene-d_8. a) non-exchangeable protons b) NH protons and carbon nuclei.

	Species	Nucleus: chemical shift / ppm																			
		Me	CHx	CHy	Meb	Nme	Hb	Ha	NH/ OH	MeC	Cu	Cv	COOH	Cmeb	CNMe	Cb	Ca	C3	C4	C2	C1
no X	T	-	-	-	0.83123	2.50199	2.81431	4.76846	nu	-	-	-	-	14.3904	34.111	60.4283	72.8966	126.12	127.075	128.123	141.423
	T2	-	-	-	0.92693	1.76305	2.24327	4.32979	nu	-	-	-	-	11.325	31.7432	59.8285	75.5834	125.738	125.95	127.097	144.265
	M	1.9717	5.6887	6.24049	-	-	-	-	-	18.8595	127.988	134.861	167.234[a]	-	-	-	-	-	-	-	-
	M2	1.95749	5.68593	6.26028	-	-	-	-	-	17.756	127.834	135.82	173.561[a]	-	-	-	-	-	-	-	-
	T*	-	-	-	0.83122	2.4819	2.79972	4.75523	3.51193	-	-	-	-	14.3389	34.0591	60.403	72.9758	126.114	127.054	128.097	141.499
	MT	nu	nu	nu	1.11419	2.73983	3.1526	5.37383	8.24926	nu	nu	nu	175.823	8.89944	31.2236	61.924	70.7434	125.783	127.341	128.281	140.234
	MT2	nu	nu	nu	1.06806	2.83536	3.54933	5.51598	10.3215	nu	nu	nu	173.306	8.69978	30.9007	61.0624	71.0399	125.71	127.478	128.368	139.347
	MT3	nu	nu	nu	1.11289	2.84072	3.51094	5.46514	13.6553	nu	nu	nu	171.26	8.39458	30.9289	61.2034	70.9692	125.675	127.311	128.201	139.918
with EDMA	M	nu	nu	nu	-	-	-	-	-	nu	nu	nu	166[b]	-	-	-	-	-	-	-	-
	M2	nu	nu	nu	-	-	-	-	-	nu	nu	nu	172[b]	-	-	-	-	-	-	-	-
	T*	-	-	-	0.85443	2.4702	2.79063	4.76141	3.60628	-	-	-	0	13.626	33.4619	60.1216	72.7709	125.718	126.462	127.562	141.766
	MT	nu	nu	nu	1.11154	2.78056	3.24756	5.4022	8.09115	nu	nu	nu	174.991	8.3902	30.5875	61.1779	70.1407	125.346	126.781	127.749	139.764
	MT2	nu	nu	nu	1.10773	2.89899	3.60332	5.50425	8.63997	nu	nu	nu	171.736	8.75056	30.7242	60.37	70.5389	125.633	126.99	127.845	139.302
	MT3	nu	nu	nu	1.11889	2.88842	3.6206	5.48246	13.619	nu	nu	nu	168.135	7.42026	30.139	60.3841	70.5472	124.378	126.733	127.642	140.342
with DVB	M	nu	nu	nu	-	-	-	-	-	nu	nu	nu	169.2[b]	-	-	-	-	-	-	-	-
	M2	nu	nu	nu	-	-	-	-	-	nu	nu	nu	173.6[b]	-	-	-	-	-	-	-	-
	T*	-	-	-	0.72219	2.31839	2.65133	4.65434	1.67206	-	-	-	0	14.1439	33.8575	60.2647	72.8248	nu	nu	nu	141.584
	MT	nu	nu	nu	0.99534	2.53365	2.9832	5.29178	8.28302	nu	nu	nu	175.606	9.09973	31.1367	61.633	70.8382	nu	nu	nu	140.411
	MT2	nu	nu	nu	1.06552	2.78865	3.60939	5.7315	9.79795	nu	nu	nu	173.261	6.67586	29.7031	61.258	70.172	nu	nu	nu	138.858
	MT3	nu	nu	nu	0.92948	2.6008	3.33005	5.29788	14.4884	nu	nu	nu	169.846	9.53897	31.3168	60.7737	71.5104	nu	nu	nu	140.336

Table S1 Fitted chemical shifts for titrations in d-chloroform. - = nucleus not present in species, nu = data not used. Shifts for T and T2 obtained from self-titration of ephedrine. Shifts for T* calculated from titration of ephedrine with MAA ignoring dimerisation of ephedrine. [a] Shifts calculated from self titration of MAA then used as constant parameters for fitting titration of ephedrine with MAA. [b] Shifts manually estimated and used as constant parameters for fitting titration of ephedrine with MAA.

	Species	Nucleus: chemical shift / ppm																			
		Me	CHx	CHy	Meb	Nme	Hb	Ha	NH/ OH	MeC	Cu	Cv	COOH	Cmeb	CNMe	Cb	Ca	C3	C4	C2	C1
no X	T	-	-	-	0.57325	2.09581	2.45462	4.60412	nu	-	-	-	-	14.0757	33.8992	60.8902	72.1366	nu	nu	nu	142.352
	T2	-	-	-	1.07	2.12695	2.61755	4.84243	nu	-	-	-	-	12.7617	33.433	61.3008	75.4931	nu	nu	nu	144.887
	M	1.57963	4.86037	5.98762	-	-	-	-	-	17.685	nu	135.727	170.365[a]	-	-	-	-	-	-	-	-
	M2	1.74956	5.27414	6.15434	-	-	-	-	-	17.7127	nu	136.272	173.942 [a]	-	-	-	-	-	-	-	-
	T*	-	-	-	0.61847	2.097	2.47196	4.62351	3.42585	-	-	-	-	13.9813	33.8463	60.9447	72.4868	nu	nu	nu	142.604
	MT	nu	nu	nu	0.96248	2.22382	2.77977	5.4339	9.04619	nu	nu	nu	175.453	8.52487	30.5826	62.2035	71.0595	nu	nu	nu	141.573
	MT2	nu	nu	nu	0.67589	2.39311	3.58838	5.64463	10.1008	nu	nu	nu	171.598	8.56199	30.728	60.8581	71.1892	nu	nu	nu	139.944

	MT3	nu	nu	nu	0.93918	2.43123	3.27785	5.50434	13.8145	nu	nu	nu	172.614	8.26632	30.6311	61.7439	71.5905	nu	nu	nu	141.032
with EDMA	M	nu	nu	nu	-	-	-	-	-	nu	nu	nu	170[b]	-	-	-	-	-	-	-	-
	M2	nu	nu	nu	-	-	-	-	-	nu	nu	nu	173[b]	-	-	-	-	-	-	-	-
	T*	-	-	-	0.73595	2.27796	2.62802	4.71268	3.46856	-	-	-	-	14.0598	33.9974	61.0884	72.9826	nu	nu	nu	142.988
	MT	nu	nu	nu	1.00719	2.46888	2.98144	5.4081	8.61182	nu	nu	nu	175.47	9.03036	30.9565	62.0264	71.0425	nu	nu	nu	141.801
	MT2	nu	nu	nu	0.98027	2.65159	3.6066	5.69823	7.55276	nu	nu	nu	172.002	8.29632	30.5834	61.175	70.7186	nu	nu	nu	140.514
	MT3	nu	nu	nu	0.96258	2.71467	3.54717	5.49472	13.8807	nu	nu	nu	169.034	8.53402	31.051	61.2583	71.8554	nu	nu	nu	141.111

Table S1 Fitted chemical shifts for titrations in d8-toluene. - = nucleus not present in species, nu = data not used. Shifts for T and T2 obtained from self-titration of ephedrine. Shifts for T* calculated from titration of ephedrine with MAA ignoring dimerisation of ephedrine. [a] Shifts calculated from self titration of MAA then used as constant parameters for fitting titration of ephedrine with MAA. [b] Shifts manually estimated and used as constant parameters for fitting titration of ephedrine with MAA.

		Nucleus: chemical shift / ppm																		
	Species	Me	CHx	CHy	Meb	Nme	Hb	Ha	NH	MeC	Cu	Cv	COOH	Cmeb	CNMe	Cb	Ca	C3	C4	C2	C1
no X	T	-	-	-	0.78647	2.43047	2.75804	4.72103	nu	-	-	-	-	13.1644	32.9467	60.0887	72.4637	125.821	126.353	127.536	142.438
	T2	-	-	-	0.9861	2.33316	2.70282	4.84027	nu	-	-	-	-	12.2515	32.7568	60.4377	73.4138	125.913	126.236	127.46	143.442
	M	1.88845	5.62794	6.04764	-	-	-	-	-	17.0569	124.963	136.015	167.308[a]	-	-	-	-	-	-	-	-
	M2	1.91327	5.7285	6.21706	-	-	-	-	-	16.7324	127.534	135.563	173.12[a]	-	-	-	-	-	-	-	-
	T*	-	-	-	0.76021	2.38861	2.71934	4.68771	3.61299	-	-	-	-	13.1584	32.9585	60.1263	72.5701	125.854	126.37	127.55	142.513
	MT	nu	nu	nu	0.97889	2.66503	3.27392	5.28619	8.39328	nu	nu	nu	174.286	8.30546	30.2488	60.7539	69.6264	125.493	126.785	127.818	140.847
	MT2	nu	nu	nu	0.98852	2.72218	3.41012	5.2551	8.71942	nu	nu	nu	171.74	8.33685	30.354	60.3348	69.8137	125.483	127.11	127.95	140.101
	MT3	nu	nu	nu	1.06859	2.86411	3.58701	5.28215	13.1895	nu	nu	nu	172.36	8.04501	30.8587	60.1789	70.8525	125.349	127.641	128.188	139.114
with EDMA	M	nu	nu	nu	-	-	-	-	-	nu	nu	nu	169.5[b]	-	-	-	-	-	-	-	-
	M2	nu	nu	nu	-	-	-	-	-	nu	nu	nu	173[b]	-	-	-	-	-	-	-	-
	T*	-	-	-	0.75966	2.39939	2.7323	4.7169	4.0939	-	-	-	-	13.0117	32.9504	60.2036	72.3637	125.795	126.324	127.522	142.415
	MT	nu	nu	nu	1.01097	2.71176	3.30347	5.33928	8.20548	nu	nu	nu	175.256	8.27426	30.2597	60.8288	69.6471	125.485	126.74	127.787	140.861
	MT2	nu	nu	nu	1.0082	2.76545	3.43707	5.30968	8.64575	nu	nu	nu	168.43	8.44274	30.4635	60.3982	69.936	125.459	126.982	127.884	140.265
	MT3	nu	nu	nu	1.12638	2.9535	3.70638	5.44702	14.0781	nu	nu	nu	166[b]	7.44818	30.5768	60.2649	70.906	125.394	127.501	128.133	139.477

Table S3 Fitted chemical shifts for titrations in d3-acetonitrile. - = nucleus not present in species, nu = data not used. Shifts for T and T2 obtained from self-titration of ephedrine. Shifts for T* calculated from titration of ephedrine with MAA ignoring dimerisation of ephedrine. [a] Shifts calculated from self titration of MAA then used as constant parameters for fitting titration of ephedrine with MAA. [b] Shifts manually estimated and used as constant parameters for fitting titration of ephedrine with MAA.