Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supporting information for

New betulin imine derivatives with antioxidant and selective antitumor activity

Manuela-Maria Iftime[1]*, Gabriela Liliana Ailiesei1[1], Sergiu Shova[1], Camelia Miron[2], Hiromasa Tanaka[2], Masaru Hori[2], Luminita Marin[1]

[1]Institute of Macromolecular Chemistry "Petru Poni", Aleea Gr. Ghica Voda, 41 A, Iasi 700487, Romania

[2]Center for Low-temperature Plasma Sciences, Nagoya University, Furo-cho, Chikusa-ku, Nagoya 464-8601, Japan

*ciobanum@icmpp.ro

FTIR spectra

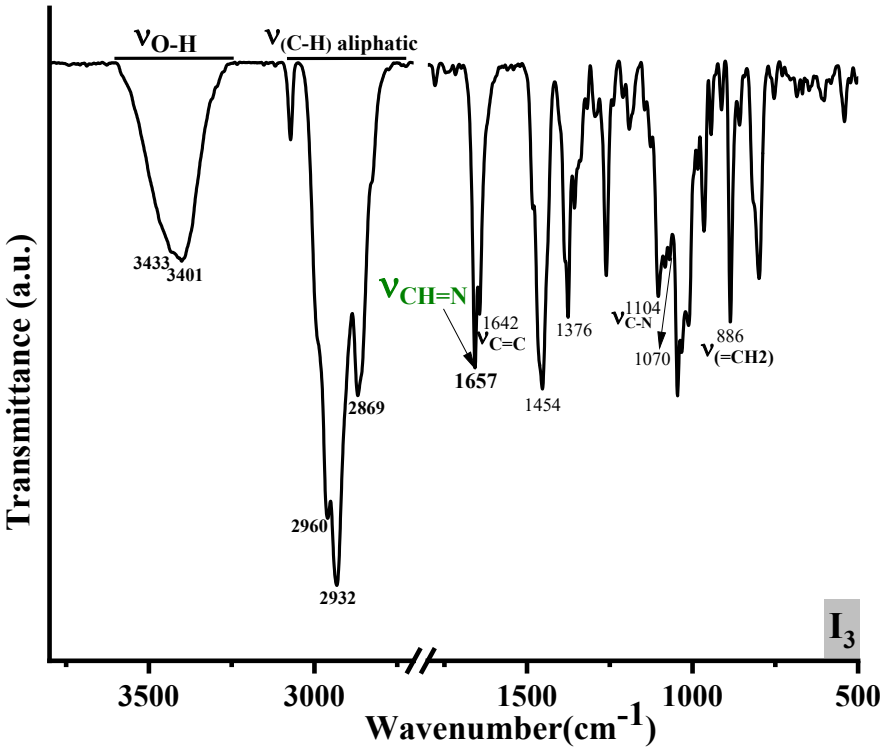

Figure 1s. FTIR spectra of **I3**

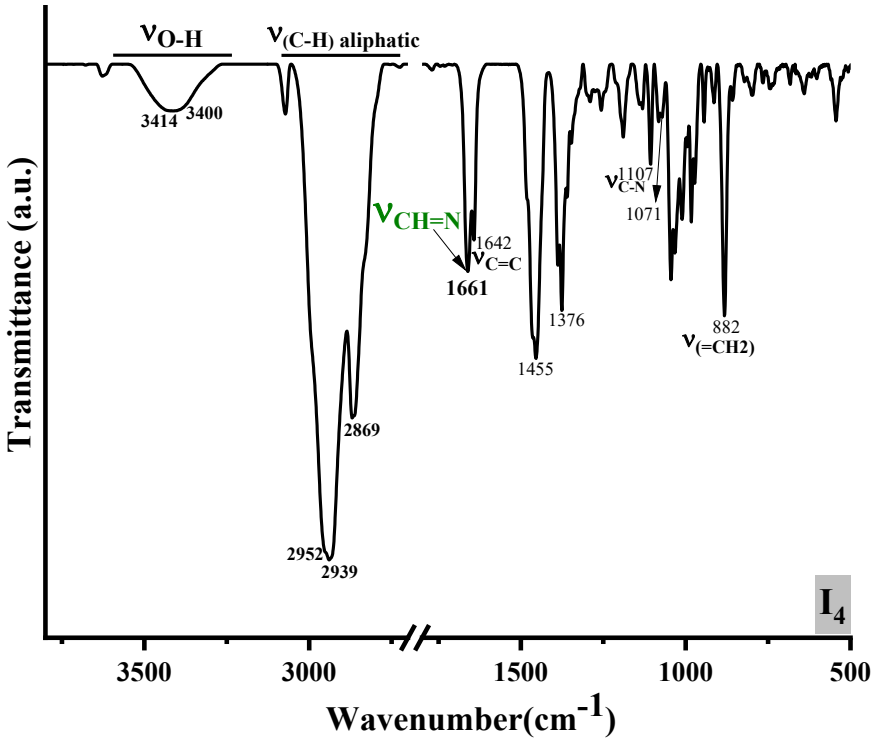

Figure 2s. FTIR spectra of $\mathbf{I_4}$

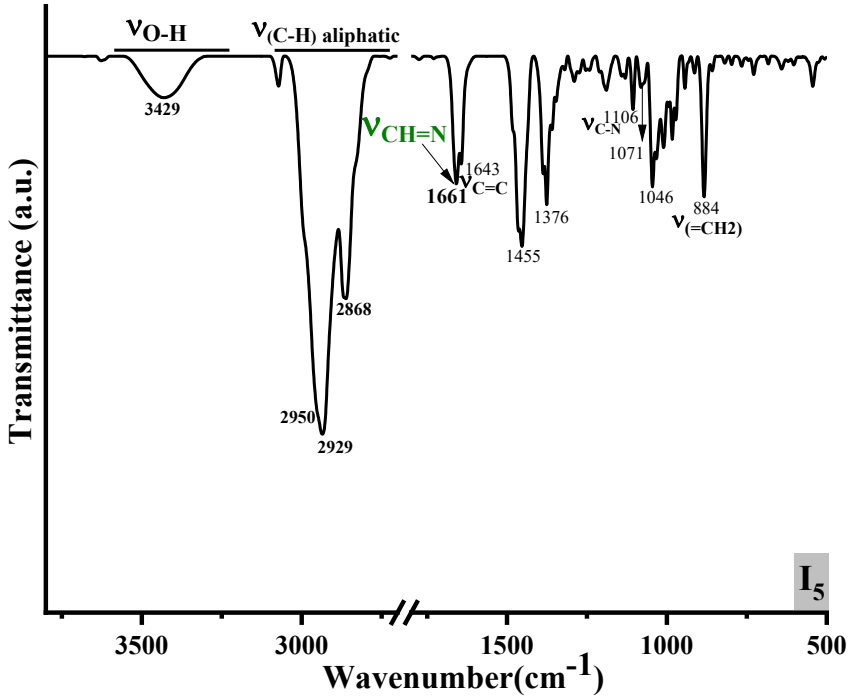

Figure 3s. FTIR spectra of $\mathbf{I_5}$

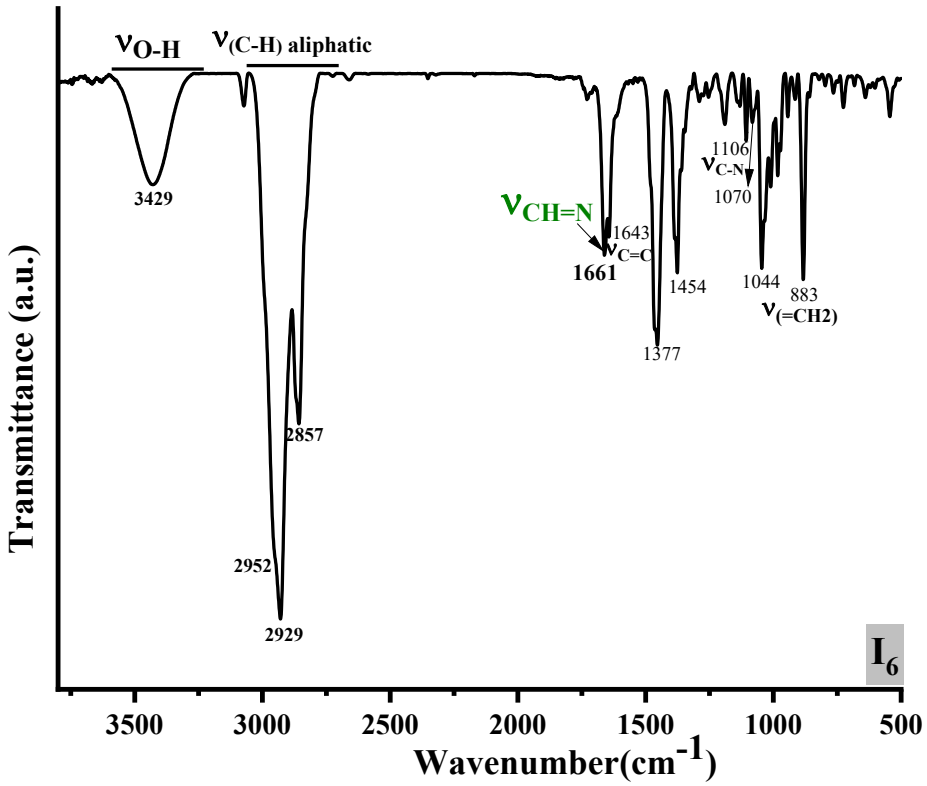

Figure 4s. FTIR spectra of $\mathbf{I_6}$

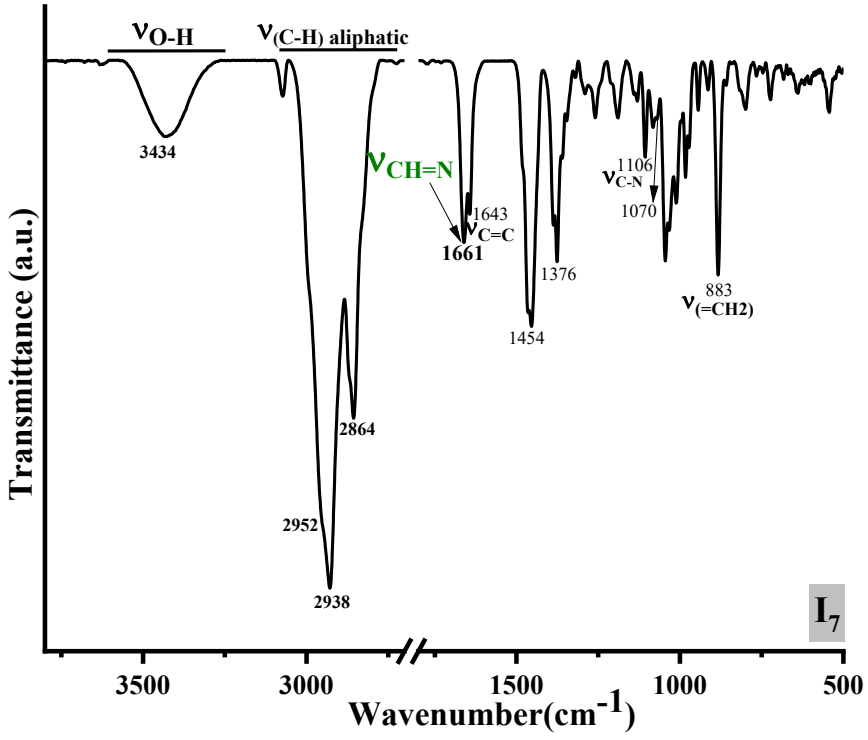

Figure 5s. FTIR spectra of $\mathbf{I_7}$

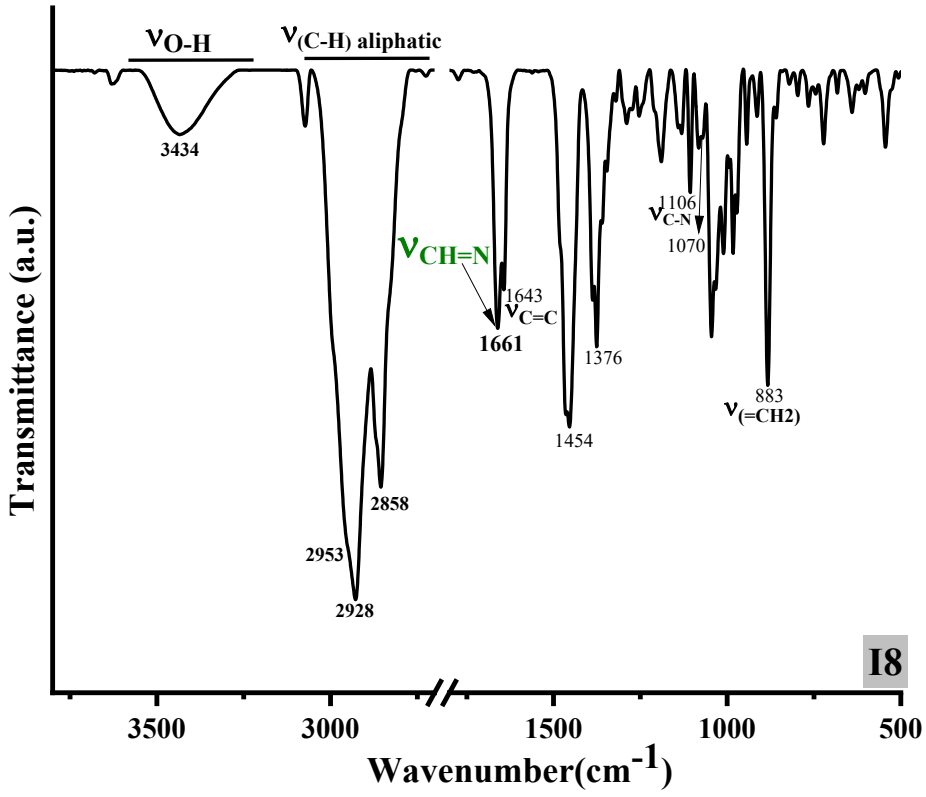

Figure 6s. FTIR spectra of $\mathbf{I_8}$

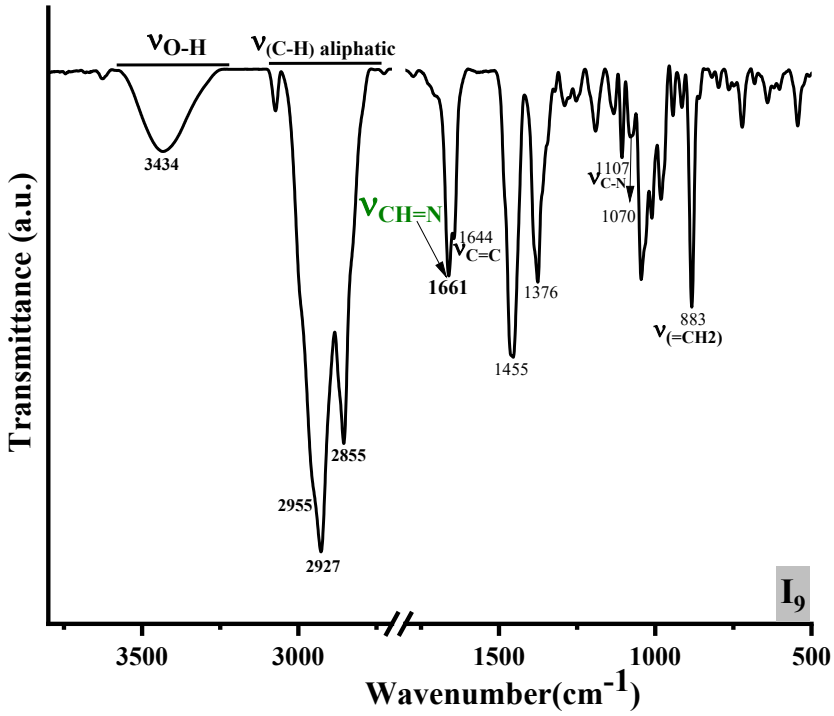

Figure 7s. FTIR spectra of $\mathbf{I_9}$

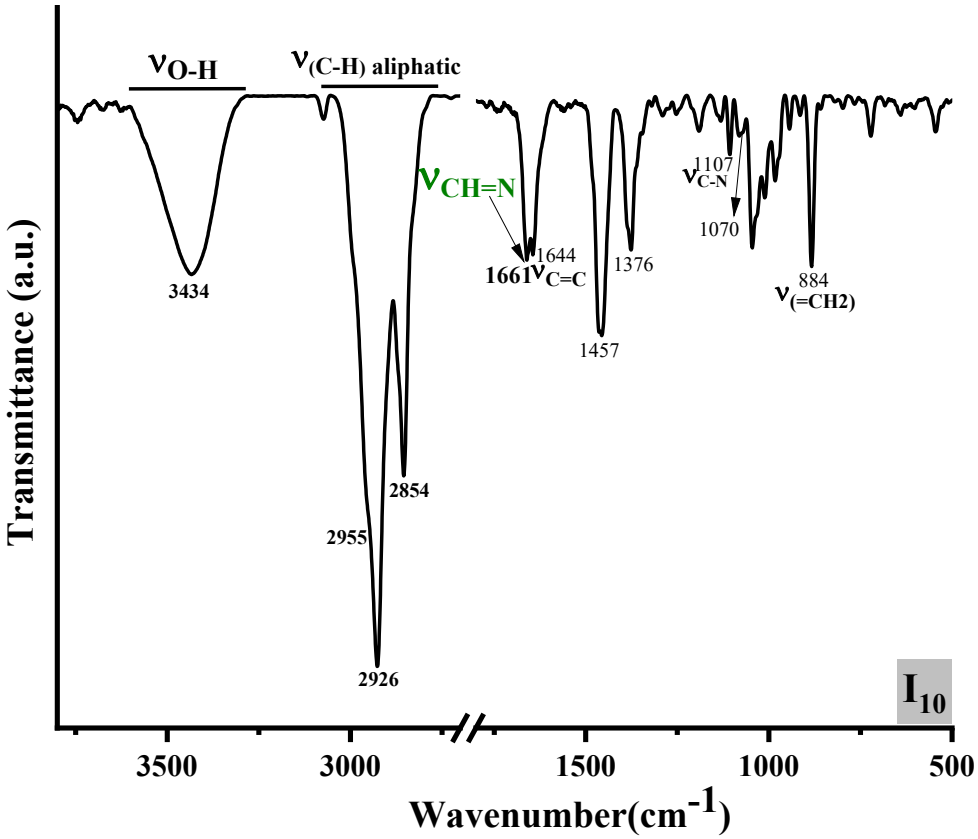

Figure 8s. FTIR spectra of $\mathbf{I_{10}}$

[1]H-NMR spectra

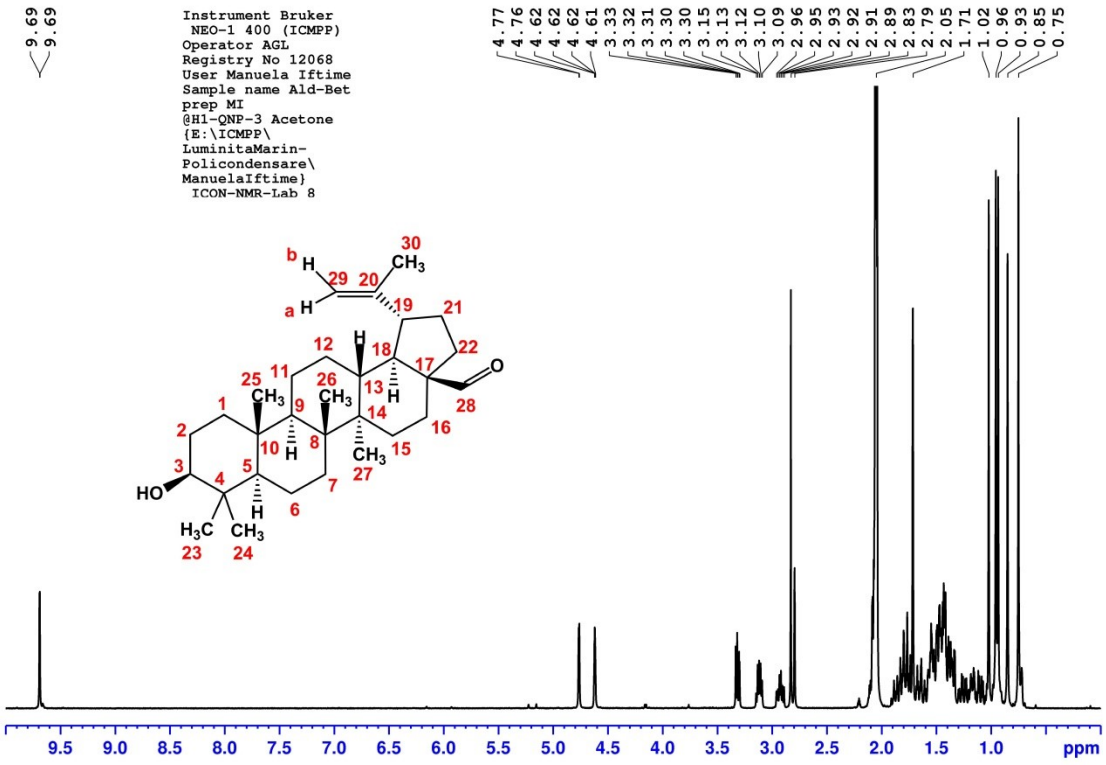

Figure 9s. [1]H NMR spectra of **Betulinic aldehyde (B)**

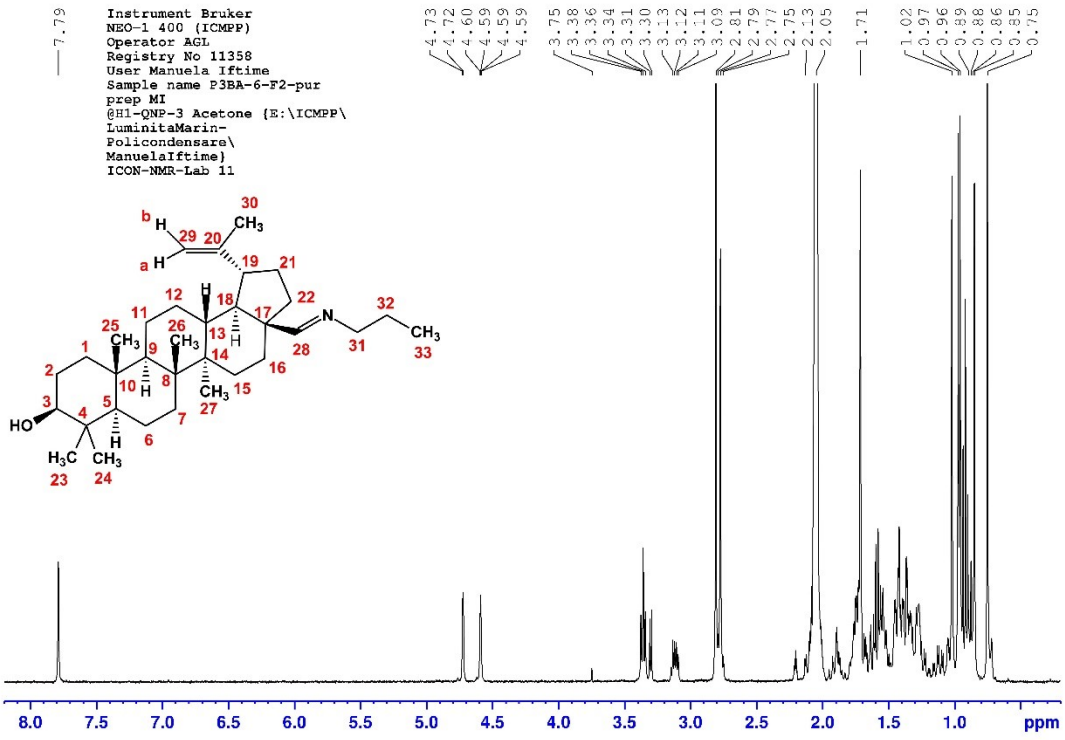

Figure 10s. ^{1}H NMR spectra of **I3**

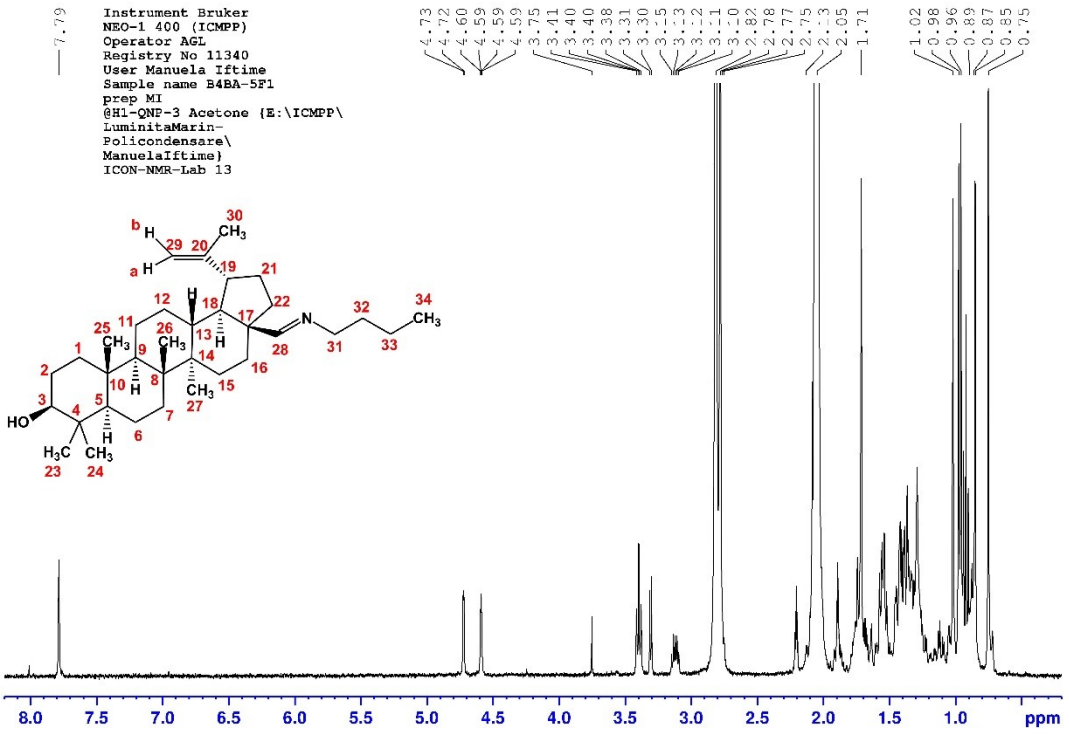

Figure 11s. ^{1}H NMR spectra of $\mathbf{I_4}$

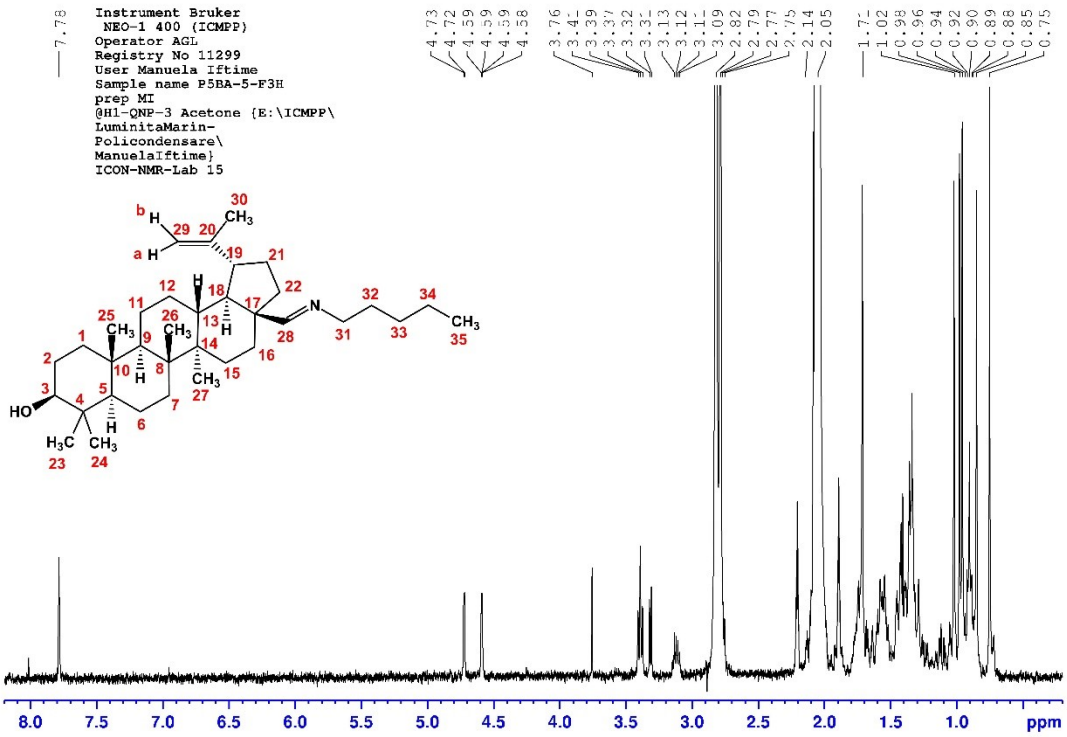

Figure 12s. ^{1}H NMR spectra of $\mathbf{I_5}$

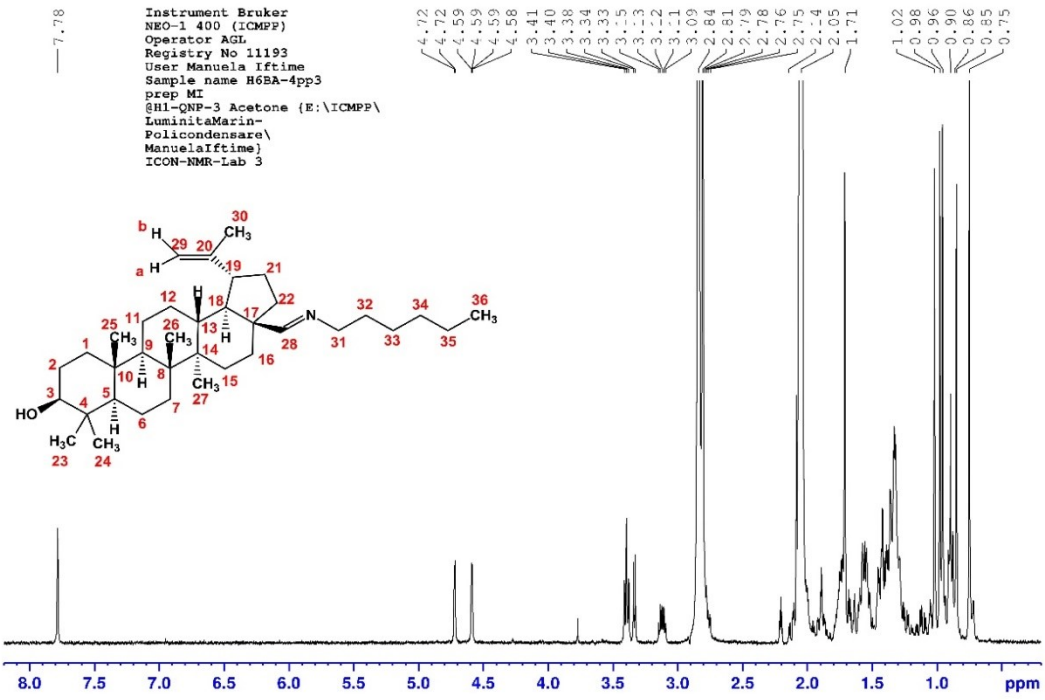

Figure 13s. ^{1}H NMR spectra of $\mathbf{I_6}$

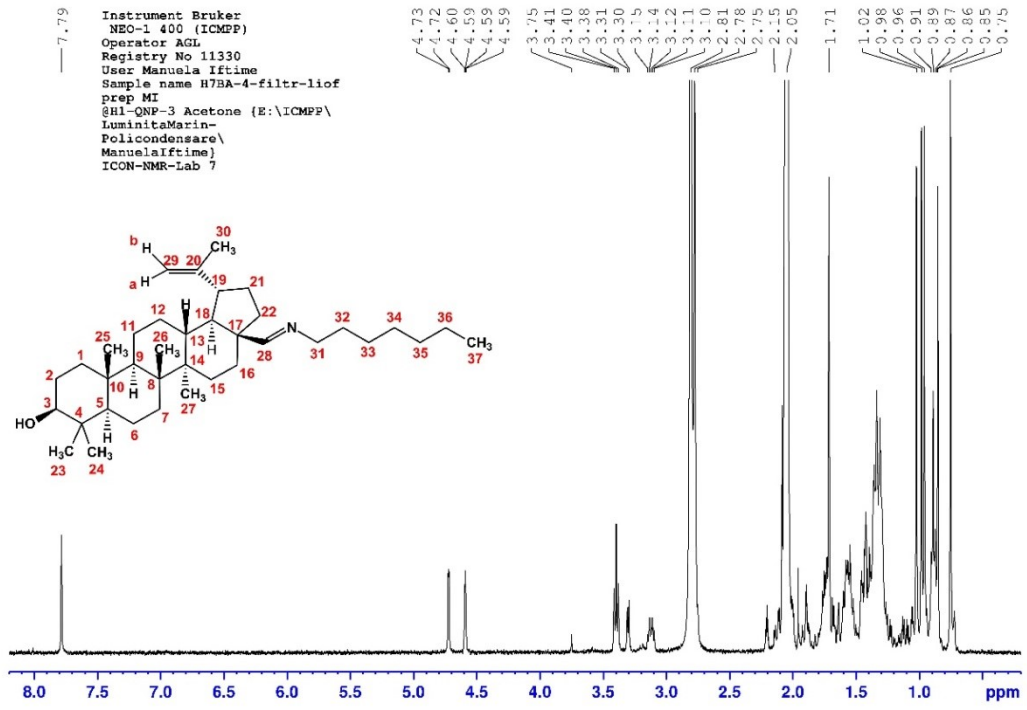

Figure 14s. ^{1}H NMR spectra of $\mathbf{I_7}$

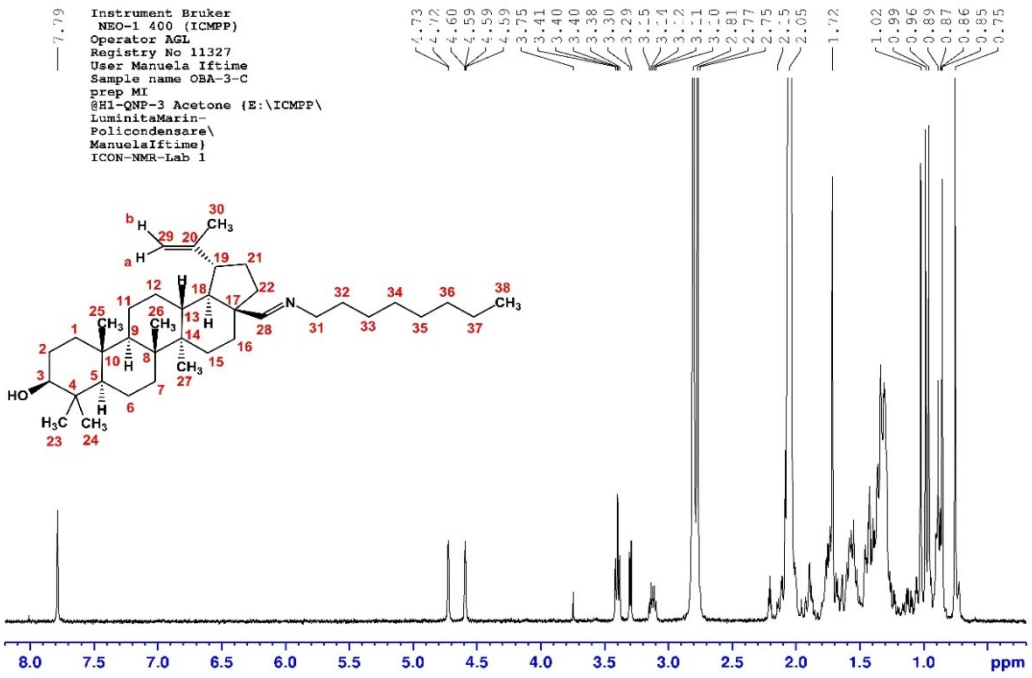

Figure 15s. ^{1}H NMR spectra of $\mathbf{I_8}$

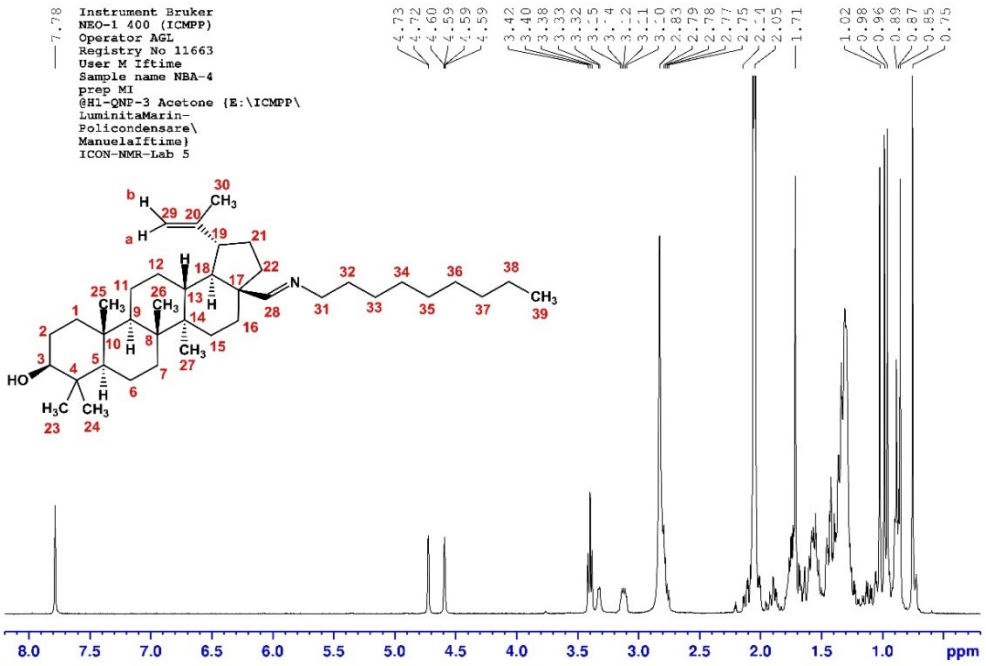

Figure 16s. ^{1}H NMR spectra of $\mathbf{I_9}$

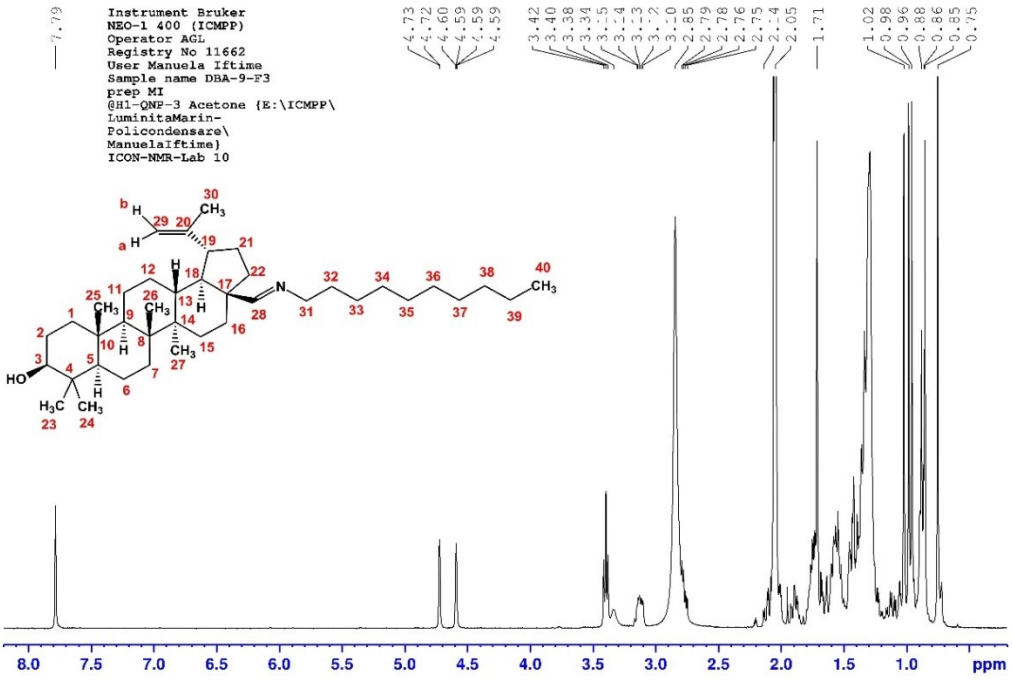

Figure 17s. ^{1}H NMR spectra of $\mathbf{I_{10}}$

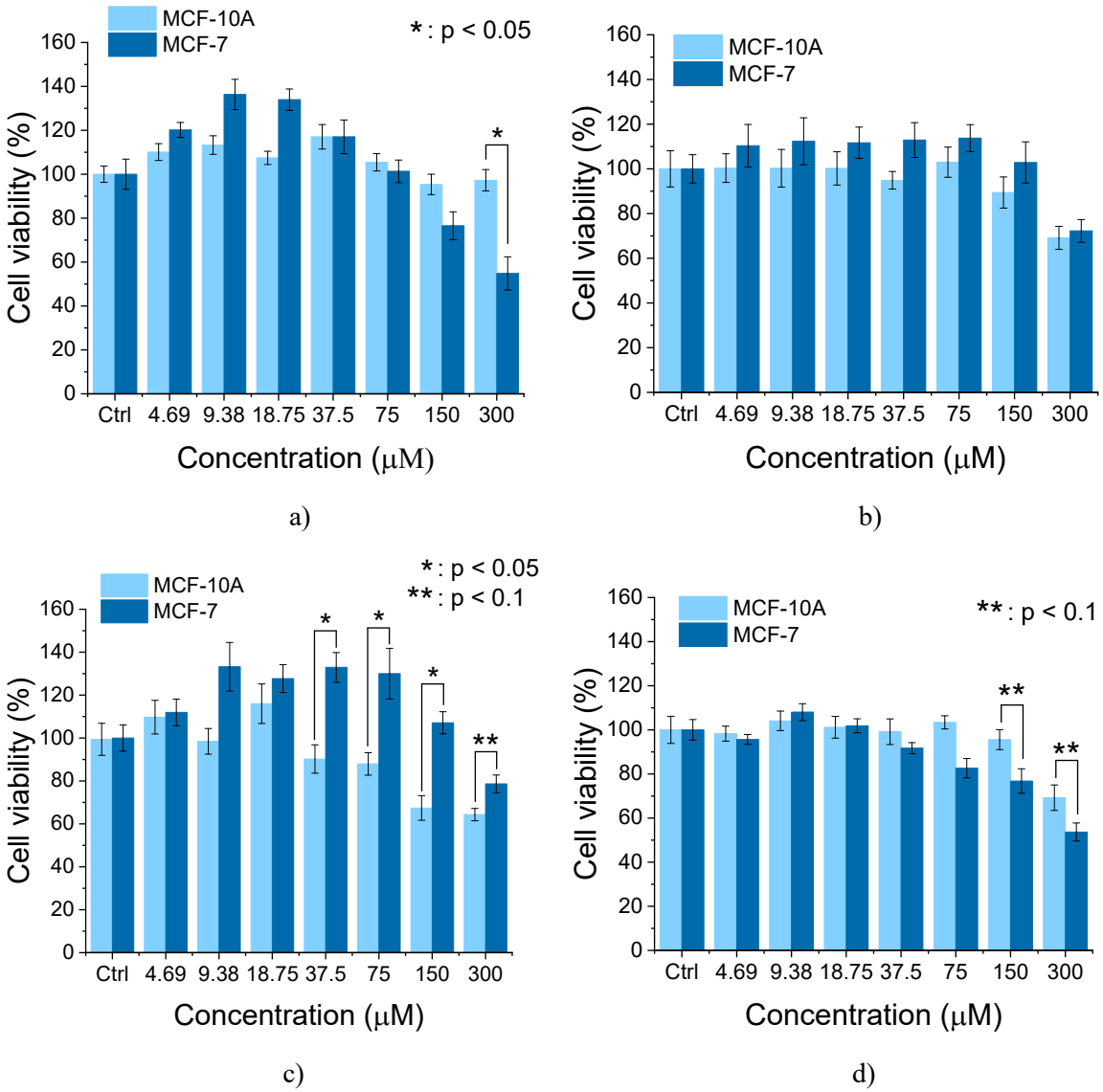

Figure 18s. MTS assay of MCF-7 and MCF-10A cells incubated with (a) I4, (b) I7, (c) I8, and (d) I10 aliphatic imines.

Table 1s. FTIR wavenumbers (cm^{-1}) and their corresponding assignment of aliphatic imines

Assignment	B	I_3	I_4	I_5	I_6	I_7	I_8	I_9	I_{10}
νO-H	3570 3474	3433 3401	3414 3400	3429	3429	3434	3434	3434	3434
νC-H assym. ($-CH_2-$) νC-H sym.($-CH_2-$)	2960sh 2938 2864	2960 2932 2869	2952 2939 2869	2950 2929 2868	2952 2929 2857	2952 2938 2864	2953 2928 2858	2955 2927 2855	2955 2926 2854
ν**CH=N**	-	1657	1661	1661	1661	1661	1661	1661	1661
ν**CH=O**	1723	-	-	-	-	-	-	-	
ν **C=C**	1641	1642	1642	1643	1643	1643	1643	1644	1644
C-H bending ($-CH_2$)	1454	1454	1455	1453	1454	1454	1454	1455	1457
νC-N	1376	1376	1376	1376	1376	1376	1376	1376	1376
νC-N str. vib.	1109 1086 -	1104 1083 1070	1107 1082 1071	1106 1081 1071	1106 1083 1070	1106 1082 1070	1106 1081 1070	1107 1081 1070	1107 1081 1070
ν ($=CH_2$)	881	886	882	884	883	883	883	884	884

Table 2s. Crystal data and details of structure refinement for compounds **B**, $\mathbf{I_3}$ and $\mathbf{I_5}$

	B 3357	**I3 6089**	**I5 6129**
Emp. formula	$C_{61}H_{100}O_5$	$C_{33}H_{55}NO$	$C_{35}H_{59}NO$
Fw	913.40	481.78	509.83
T [K]	200	293(2)	293(2)
space group	$P2_12_12_1$	$P2_12_12_1$	$P2_12_12$
a [Å]	7.0481(6)	6.9924(8)	37.2840(16)
b [Å]	25.4221(9)	12.499(2)	12.3609(5)
c [Å]	30.6301(14)	34.223(4)	6.9822(3)
V [Å^3]	5488.3(6)	2991.0(7)	3217.8(2)
Z	4	4	4
ρ_{calcd} [g cm^{-3}]	1.105	1.070	1.052
μ [mm^{-1}]	0.068	0.062	0.061
Crystal size [mm]	0.10 × 0.15 × 0.60	0.15 × 0.05 × 0.05	0.25 × 0.15 × 0.15
2Θ range	3.104 to 50.05	3.47 to 50.054	3.472 to 50.048
Refls. collected	24746	13161	12440
Indep. Refls., R_{int}	9535, 0.0412	5286, 0.0845	7245, 0.0239
Data/rests./params.	9535/0/609	5286/0/324	7245/3/327
GOF	1.102	0.988	0.993
R_1, wR_2(all data)	0.0735, 0.1390	0.0843, 0.1557	0.0822, 0.2361
CCDC no.	**2257742**	**2257743**	**2257744**

Table 3s. Solubility of the aliphatic imines in common solvents

Solvent	δ	I_3	I_4	I_5	I_6	I_7	I_8	I_9	I_{10}
Water	23.43	–	–	–	–	–	–	–	–
Methanol	14.48	+	+	+	+	+	+	+	+
Ethanol	12.98	+	+	+	+	+	+	+	+
n-butanol	11.32	+	+	+	+	+	+	+	+
Dimethylsulfoxide	13.04	+	+	+	+	+	+	+	+
Dichloromethane	9.9	+	+	+	+	+	+	+	+
Acetone	9.75	+	+	+	+	+	+	+	+
Acetonitrile	15.3	+	+	+	+	+	+	+	+
Ethyl acetate	9.1	+	+	+	+	+	+	+	+
Hexane	7.24	+	+	+	+	+	+	+	+

+: soluble at room temperature; -: insoluble.

[a] Solvent solubility parameter $(cal\ cm^{-3})^{1/2}$

Table 4s. Thermal behavior of the studied aliphatic imine and betulinic aldehyde

Code	T_m [a]	$T_{5\%}$ [b]	$T_{10\%}$ [c]	T_{dec} [d]
B	167-170	227	257	310, 394
I_3	129-133	250	261	302
I_4	120-122	-	-	-
I_5	116-123	252	263	312
I_6	105-113	-	-	
I_7	76-80	262	278	328
I_8	50-66	-	-	-
I_9	46-57	-	-	-
I_{10}	42-50	-	-	-

[a] $T_{m\%}$ = melting temperature and softening temperature range determined by POM;

[b] $T_{5\%}$ = temperature corresponding to 5% mass loss;

[c] $T_{10\%}$ = temperature corresponding to 10% mass loss;

[d] T_{dec} = temperature corresponding to the maximum degradation rate