1-Chloro-10-Undecene.\(^1\) A mixture of 25.0 g (0.147 mol) of 10-undecylenyln alcohol, 50.0 g (0.190 mol) of triphenylphosphine, 75 ml of carbon tetrachloride, and 75 ml of acetonitril was heated to reflux for 24 h. The resulting brown solution was cooled to room temperature and the solvents were evaporated under reduced pressure. The solid residue was transferred onto a glass filter and extracted four times with 100 ml of petroleum ether (40–60 °C). The combined extracts were dried over MgSO\(_4\) and concentrated in vacuo, which yielded 29.0 g of crude 1-chloro-10-undecene as a yellow oil.\(^2\) Distillation gave 24.8 g (0.131 mol, 89%) of pure 1-chloro-10-undecene (bp 115–116 °C, 13 mmHg; lit 104–105 °C, 6 mmHg).\(^3\)

\(^1\)H NMR: \(\delta\) 5.90–5.70 (m, 1H), 5.04–4.88 (m, 2H), 3.52 (t, 2H, \(J = 6.9\) Hz), 2.08–1.98 (m, 2H), 1.82–1.69 (m, 2H), 1.50–1.22 (m, 12H). \(^13\)C NMR: \(\delta\) 139.15, 114.14, 45.14, 33.81, 32.67, 29.42, 29.39, 29.10, 28.92, 28.89, 26.89. IR (cm\(^{-1}\)): 3076 (m); 2926 (m); 2854 (m); 1824 (w); 1640 (m); 1463 (m); 1445 (w). MS: m/z (relative intensity) 41 (72.4); 55 (100.0); 69 (68.4); 83 (38.5); 97 (26.3); 104 (50.8); 188 (10.4); 190 (2.8). Exact mass: calculated 188.358882 for \(\text{C}_{11}\text{H}_{22}\text{Cl}\); found 188.3531.

\(N\)-(ω-Undecylenyl)-Phthalimide (I).\(^4\) To a solution of 10.0 g (0.053 mol) of 1-chloro-10-undecene in 25 ml of dry DMF was added 12.8 g (0.069 mol) of potassium phthalimide. The resulting suspension was stirred on an oil bath of 90 °C for 24 h. The obtained reaction mixture was cooled to room temperature and 75 ml of water was added. The aqueous layer was extracted once with 75 ml and subsequently twice with 30 ml of ether. The combined organic layers were washed with 25 ml of a 0.2 M NaOH solution and with 25 ml of brine, and dried over MgSO\(_4\). Evaporation of the solvent yielded 15.5 g (0.052 mol, 98%) of the crude product as a yellow solid. Recrystallization from 50 ml of distilled methanol gave, after standing of the solution at 18–20 °C in a closed flask for 18 h, 10.3 g (0.034 mol, 64%) of \(N\)-(ω-undecylenyl)-phthalimide (I) as white needles (mp 42–42.5 °C). The filtrate was subsequently placed at 4 °C in a closed flask for 24 h, which yielded an additional 2.6 g of I, which was still pure enough to use. This gave a total yield of 12.9 g (0.043 mol, 83%).

\(^1\)H NMR: \(\delta\) 7.86–7.80 (m, 2H), 7.75–7.67 (m, 2H), 5.90–5.71 (m, 1H), 5.04–4.89 (m, 2H), 3.67 (t, 2H, \(J = 7.3\) Hz), 2.06–1.97 (m, 2H), 1.72–1.58 (m, 2H), 1.41–1.19 (m, 12H). \(^13\)C NMR: \(\delta\) 168.37 (2C), 139.12, 133.75 (2C), 132.10 (2C), 123.06 (2C), 114.04, 37.99, 33.72, 29.34, 29.31, 29.09, 29.00, 28.82, 28.53, 26.78. IR (CCl\(_4\), cm\(^{-1}\)): 3079 (w); 2929 (m); 2856 (m); 1774 (m); 1718 (s).
1-Amino-10-Undecene. To a solution of 10.0 g (0.033 mol) of \( N-(\omega\text{-undecylenyl}) \)-phthalimide (I) in 100 ml of ethanol was added 2.5 g (0.050 mol) of hydrazine. The resulting mixture was heated to reflux for 3 h. The solution was cooled to room temperature and acidified to \( \text{pH} = 1-2 \) by the addition of 100 ml of 1 M HCl. The white suspension was filtered on a glass filter and the residue was washed twice with 20 ml of 1 M HCl. The combined filtrates were made alkaline (\( \text{pH} = 10-11 \)) by the addition of NaOH (tablets) and concentrated to a volume of ca. 100 ml by evaporation under reduced pressure. The resulting turbid aqueous layer was extracted four times with 50 ml of ether. The combined organic layers were washed once with 20 ml of a 0.2 M NaOH solution and once with 20 ml of brine, to which a few ml of the 0.2 M NaOH solution has been added. The organic layer was dried over NaOH (solid) for 1–2 h. Evaporation of the solvent yielded 4.9 g (0.029 mol, 88%) of crude 1-amino-10-undecene as a yellow oil.

\(^1\text{H NMR: } 8 5.92-5.72 (m, 1H), 5.04-4.90 (m, 2H), 2.68 (t, 2H, \( J = 6.8 \text{ Hz} \)), 2.09-1.99 (m, 2H), 1.74 (br. s, 2H), 1.45-1.27 (m, 14H). \(^{13}\text{C NMR: } 8 139.19, 114.06, 42.09, 33.76, 33.55, 29.51, 29.42, 29.39, 29.08, 28.88, 26.83. \text{ IR (cm}^{-1}): 3366 \text{ (m); 3295 (m); 3076 (w); 2925 (m); 2853 (m); 1640 (m).}

\( N-(\omega\text{-Undecylenyl})\)-Acetamide (II). A mixture of 4.2 g (0.025 mol) of 1-amino-10-undecene and 5 ml (0.053 mol) of acetic anhydride was heated to reflux for 10 min, using a preheated oil bath of 170 °C. The resulting hot solution was immediately poured onto 50 ml of ice water and this mixture was heated to reflux for 20 min. The resulting solution was cooled to room temperature and extracted three times with 50 ml of ether. The combined organic layers were subsequently washed twice with water and once with brine and dried on MgSO₄. Evaporation of the solvent gave 5.0 g of a yellow oil, which contained a mixture (ratio ~2 : 1) of the monoacetylated amine (i.e., compound II) and the diacetylated amine.

The compounds were separated by chromatography (silica gel) using a 2 : 1 mixture of distilled ethyl acetate and distilled petroleum ether (40–60 °C). The fractions containing II were combined and stirred with a small amount of activated carbon (Norit) for 18 h. The resulting solution was dried over MgSO₄ and filtered. Subsequent evaporation of the solvents under reduced pressure gave 2.7 g (0.013 mol, 52%) of \( N-(\omega\text{-undecylenyl})\)-acetamide (II) as a white solid (mp 29–30 °C). Subsequent recrystallization from 25 ml of distilled ethyl acetate at -20 °C gave 0.90 g (0.0042 mol, 16%) of pure \( N-(\omega\text{-undecylenyl})\)-acetamide (II) as white crystals (mp 30–31 °C).
Amino-terminated organic monolayers on hydrogen-terminated silicon surfaces

$^1$H NMR: $\delta$ 5.90–5.70 (m, 1H), 5.50 (br. s, 1H), 5.02–4.88 (m, 2H), 3.26–3.16 (m, 2H), 2.05–1.96 (m, 2H), 1.96 (s, 3H), 1.50–1.22 (m, 14H). $^{13}$C NMR: $\delta$ 170.14, 139.15, 114.08, 39.71, 33.75, 29.51, 29.42, 29.35, 29.22, 29.04, 28.85, 26.87, 23.22. IR (KBr, cm$^{-1}$): 3292 (m, br. peak); 3079 (w); 2928 (m); 2855 (m); 1650 (s); 1553 (m). MS: m/z (relative intensity) 30 (100); 41 (24.6); 43 (33.8); 55 (19.49); 60 (30.4); 72 (50.5); 73 (51.6); 86 (22.3); 100 (20.8); 211 (16.6). Exact mass: calculated 211.1936 for C$_{13}$H$_{23}$NO; found 211.1939.

Cassie plot of the monolayers of I (series 1 & 2) and II (series 3 & 4), respectively before and after reaction with hydrazine solution (so series 1 & 3 are before reaction, and 2 & 4 after reaction with hydrazine solution). Cassie has derived the equation $\cos \theta_{\text{bin}} = \chi_1 \cos \theta_1 + \chi_2 \cos \theta_2$, in which $\theta_{\text{bin}}$, $\theta_1$ and $\theta_2$ are the contact angles of a binary surface consisting of components 1 and 2, and the contact angles of the surfaces of pure 1 and 2, and in which $\chi$ represents the fractional surface coverage of components 1 and 2, respectively. From ca. 5% of I/II in solution onwards, the Cassie plots (y-axis: $\cos \theta_{\text{bin}}$, x-axis: percentage of I/II in solution) are approximately linear. This point to a 1:1 correspondence between the solution and surface compositions.


2 The crude material usually contains a small amount of triphenylphosphine and/or triphenylphosphine oxide, thus the apparent yield is always > 100%.


7 If this step with activated carbon (Norit) is omitted, the obtained product (II) is pale yellow instead of white. It should also be noted that the product is not the first to come of the column, which somewhat complicates this purification by chromatography.