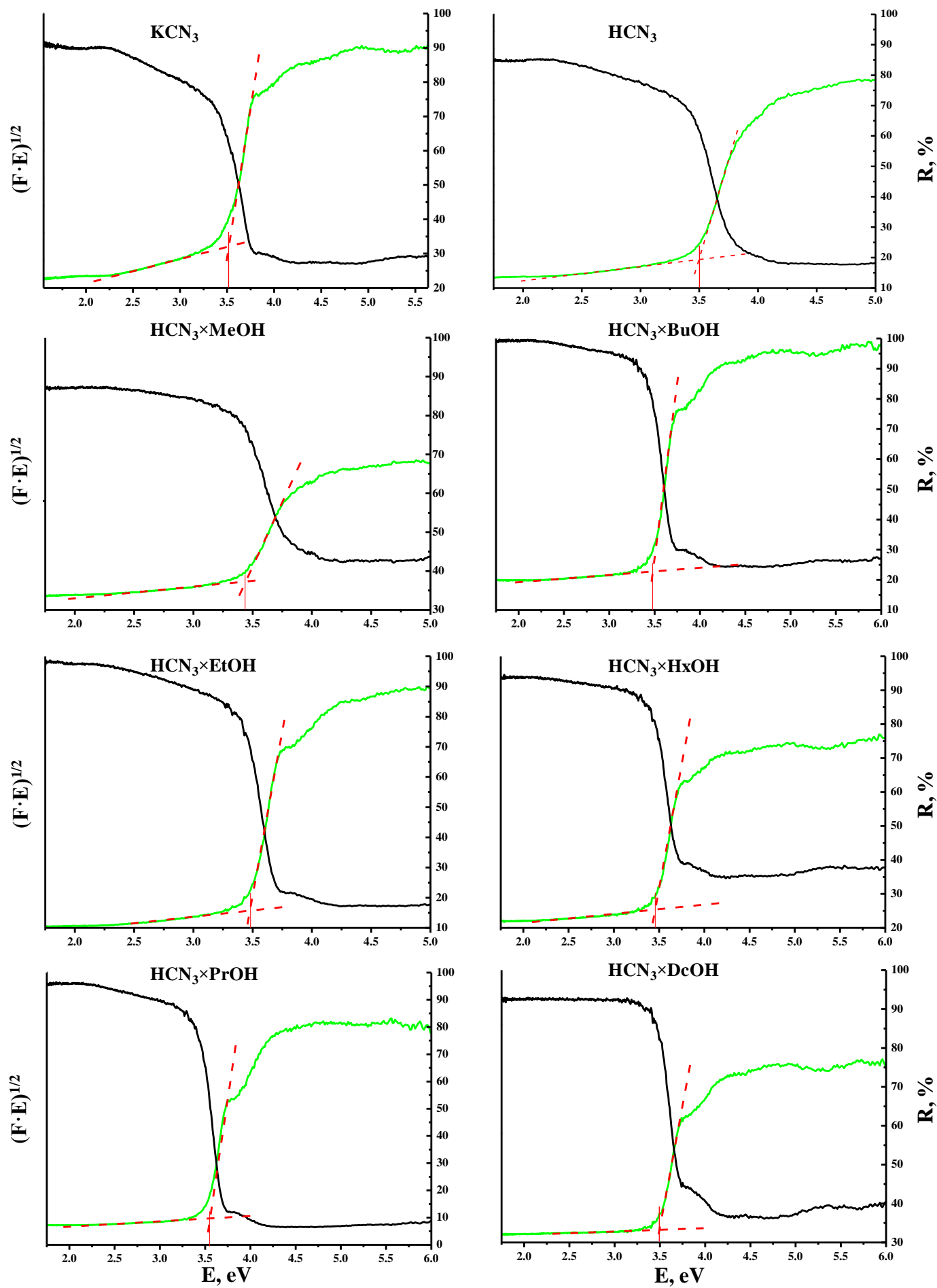# Supporting Information S1

Diffuse reflectance spectra and Tauc plots for initial niobates and their *n*-alkoxy derivatives
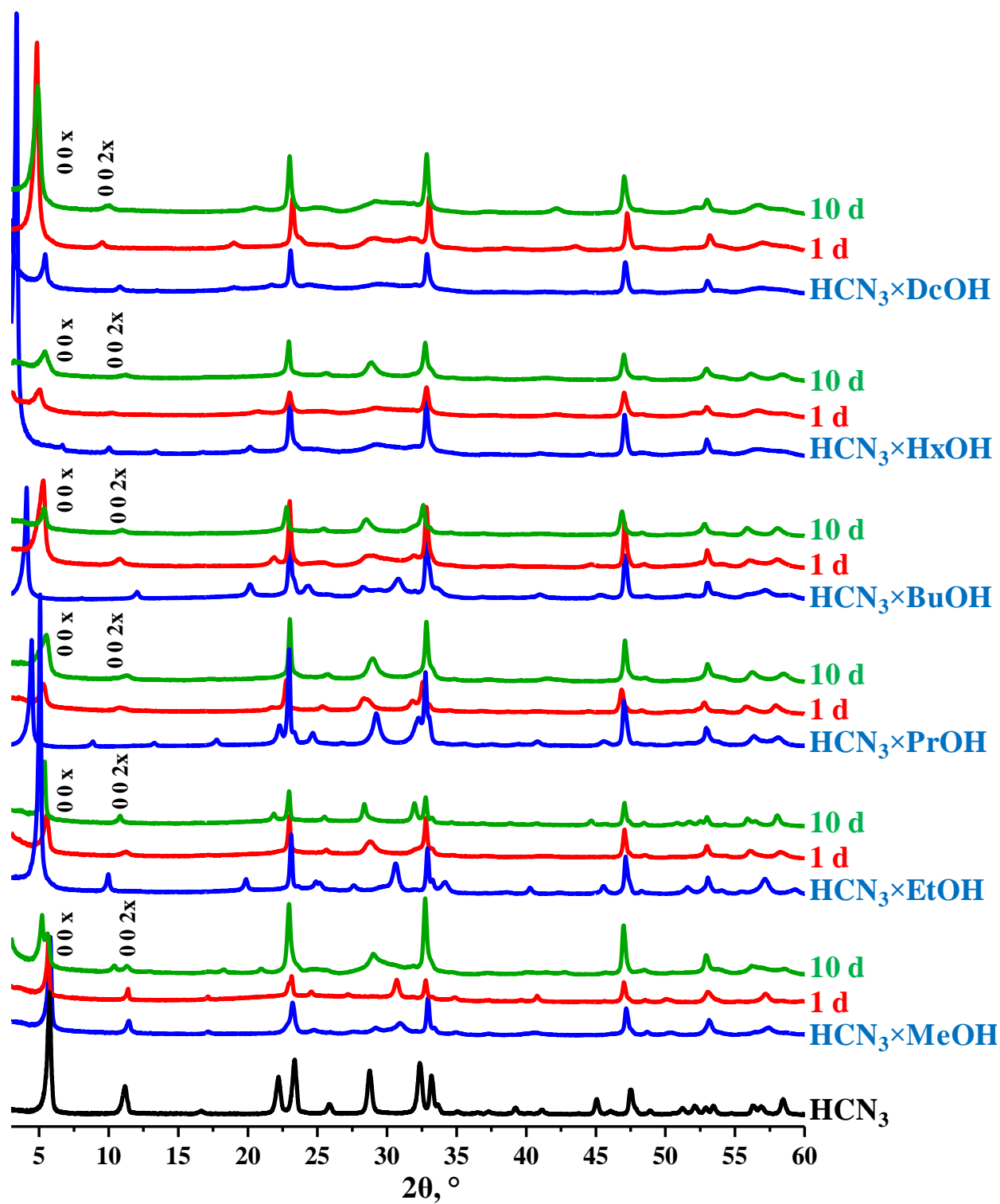
Supporting Information S2

Quantitative compositions *n*-alkoxy derivatives before and after keeping under reduced pressure

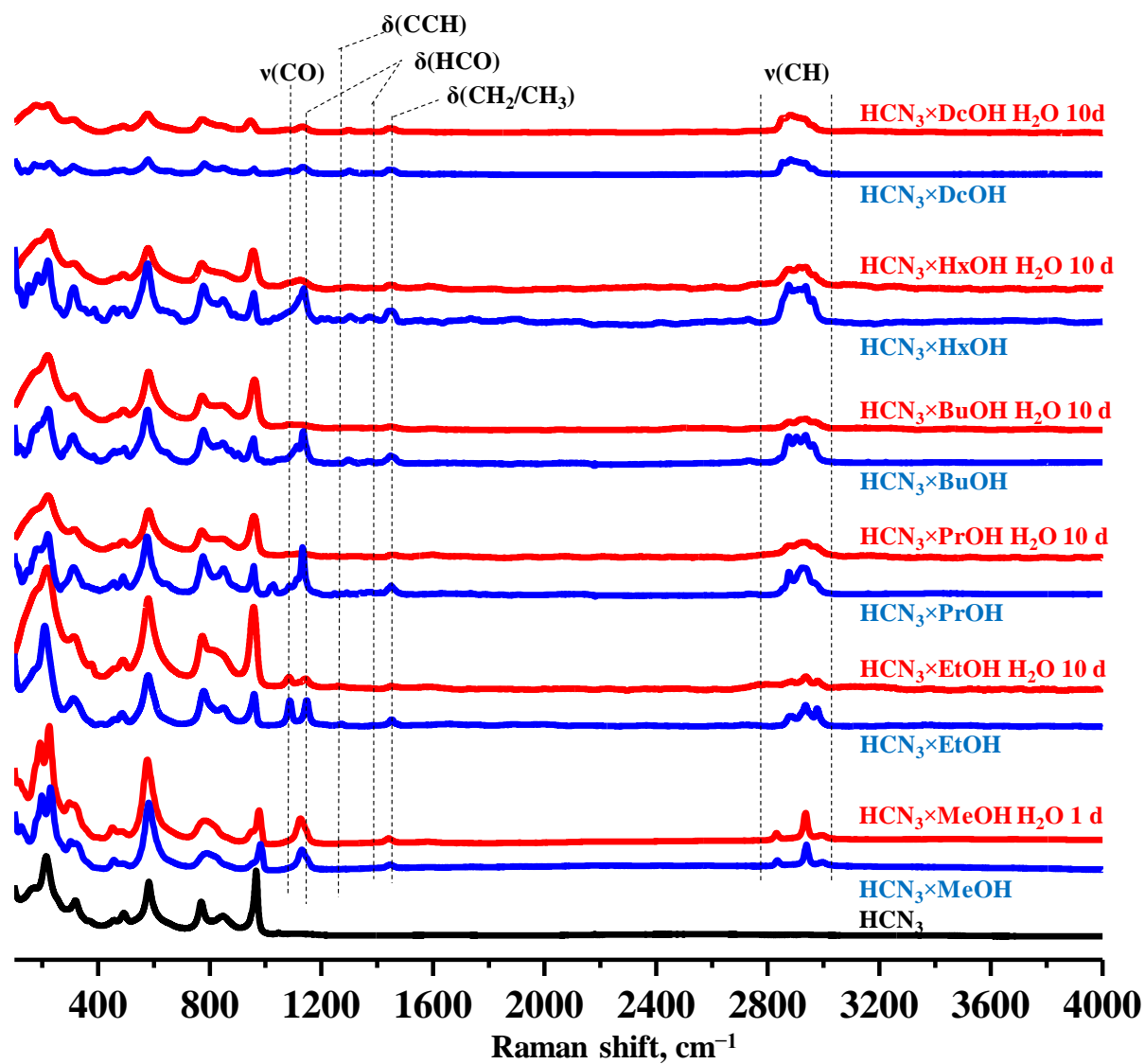| Sample	Organic content (x)		
	initial	5 d vacuum	10 d vacuum
$HCN_3 \times MeOH$	0.99	0.97	0.95
$HCN_3 \times EtOH$	0.82	0.66	0.64
$HCN_3 \times PrOH$	0.86	0.78	0.74
$HCN_3 \times BuOH$	0.86	0.77	0.76
$HCN_3 \times HxOH$	0.86	0.79	0.78
$HCN_3 \times DcOH$	0.90	0.70	0.70

XRD patterns of *n*-alkoxy derivatives before and after water treatment

Supporting Information S4

Raman spectra of *n*-alkoxy derivatives before and after water treatment
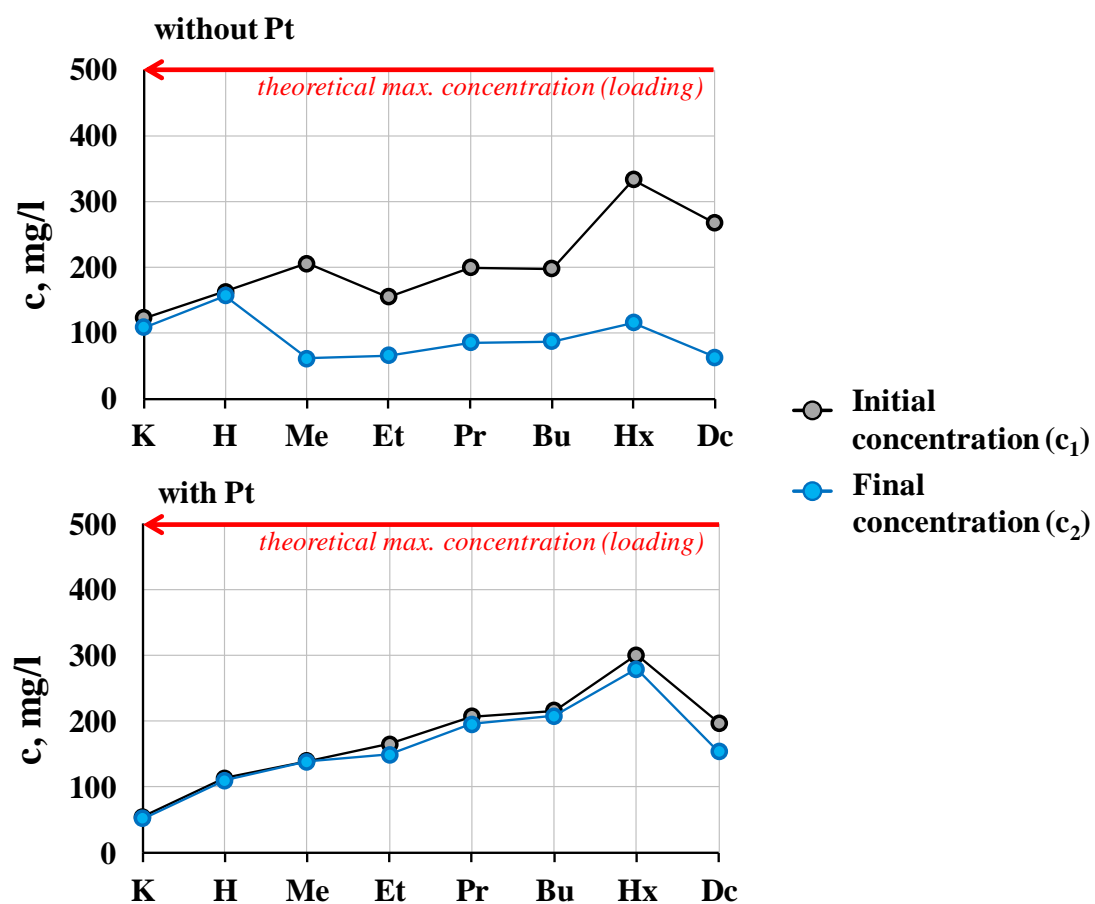
Supporting Information S5

Actual volume concentrations and pH values of reaction suspensions

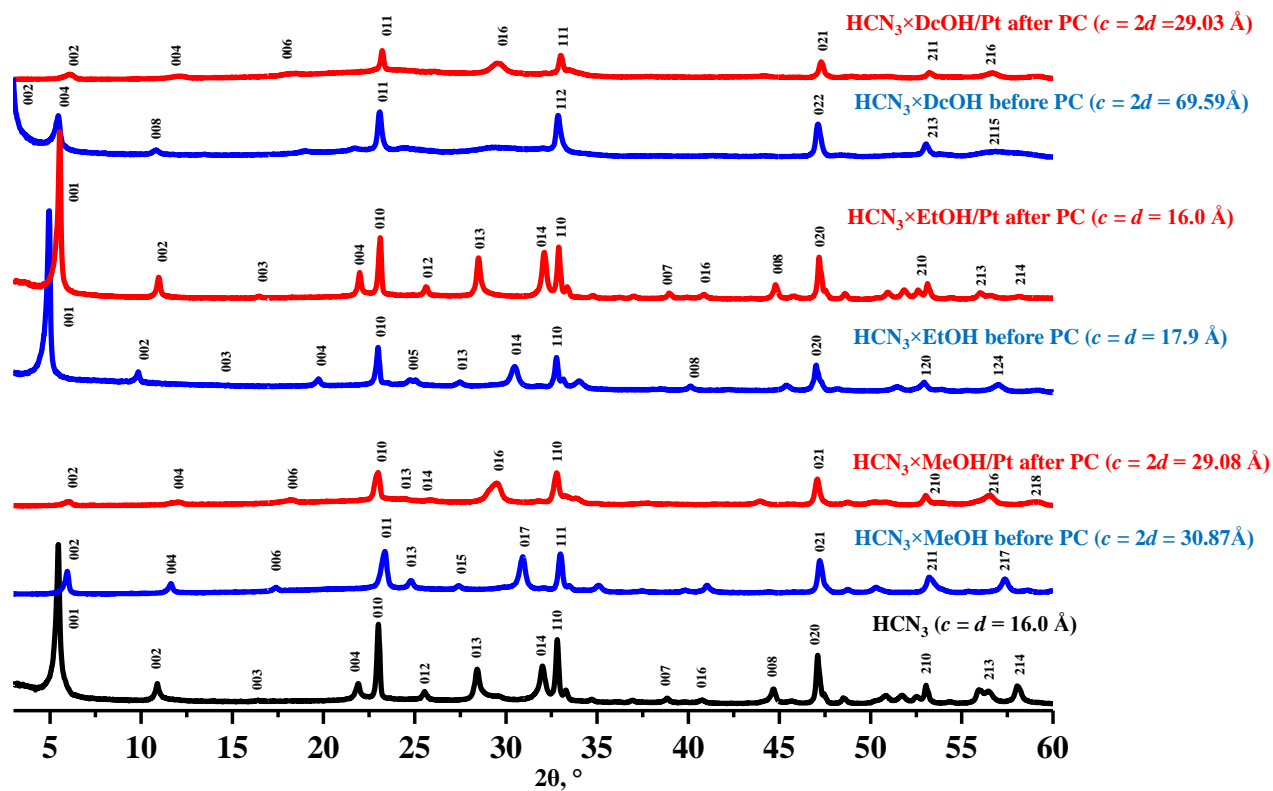| Photocatalyst	c_1, mg/l	c_2, mg/l	c_3, mg/l	pH_1	pH_2	pH_3
KCN_3	123	109	0.16	6.6	7.1	7.1
KCN_3/Pt	54.0	51.5	–	5.3	4.8	4.5
HCN_3	163	157	0.00	4.2	4.0	3.9
HCN_3/Pt	113	110	–	3.5	3.2	3.2
$HCN_3\times MeOH$	206	61.4	0.04	4.3	3.8	3.7
$HCN_3\times MeOH$/Pt	139	138	–	3.7	3.3	3.4
$HCN_3\times EtOH$	155	65.8	1.70	4.3	3.8	3.8
$HCN_3\times EtOH$/Pt	165	149	–	3.7	3.2	3.2
$HCN_3\times PrOH$	200	85.6	0.09	4.3	4.0	4.1
$HCN_3\times PrOH$/Pt	207	195	–	3.3	3.5	3.5
$HCN_3\times BuOH$	198	87.6	0.07	4.3	4.0	4.0
$HCN_3\times BuOH$/Pt	216	208	–	3.8	3.5	3.5
$HCN_3\times HxOH$	333	116	0.15	4.3	3.9	3.9
$HCN_3\times HxOH$/Pt	300	279	–	3.9	3.5	3.6
$HCN_3\times DcOH$	267	63.1	0.17	4.1	3.9	3.9
$HCN_3\times DcOH$/Pt	197	153	–	3.9	3.6	3.6

Comparison of actual concentrations of photocatalysts in the reaction suspensions in the beginning and in the ending of photocatalytic measurements
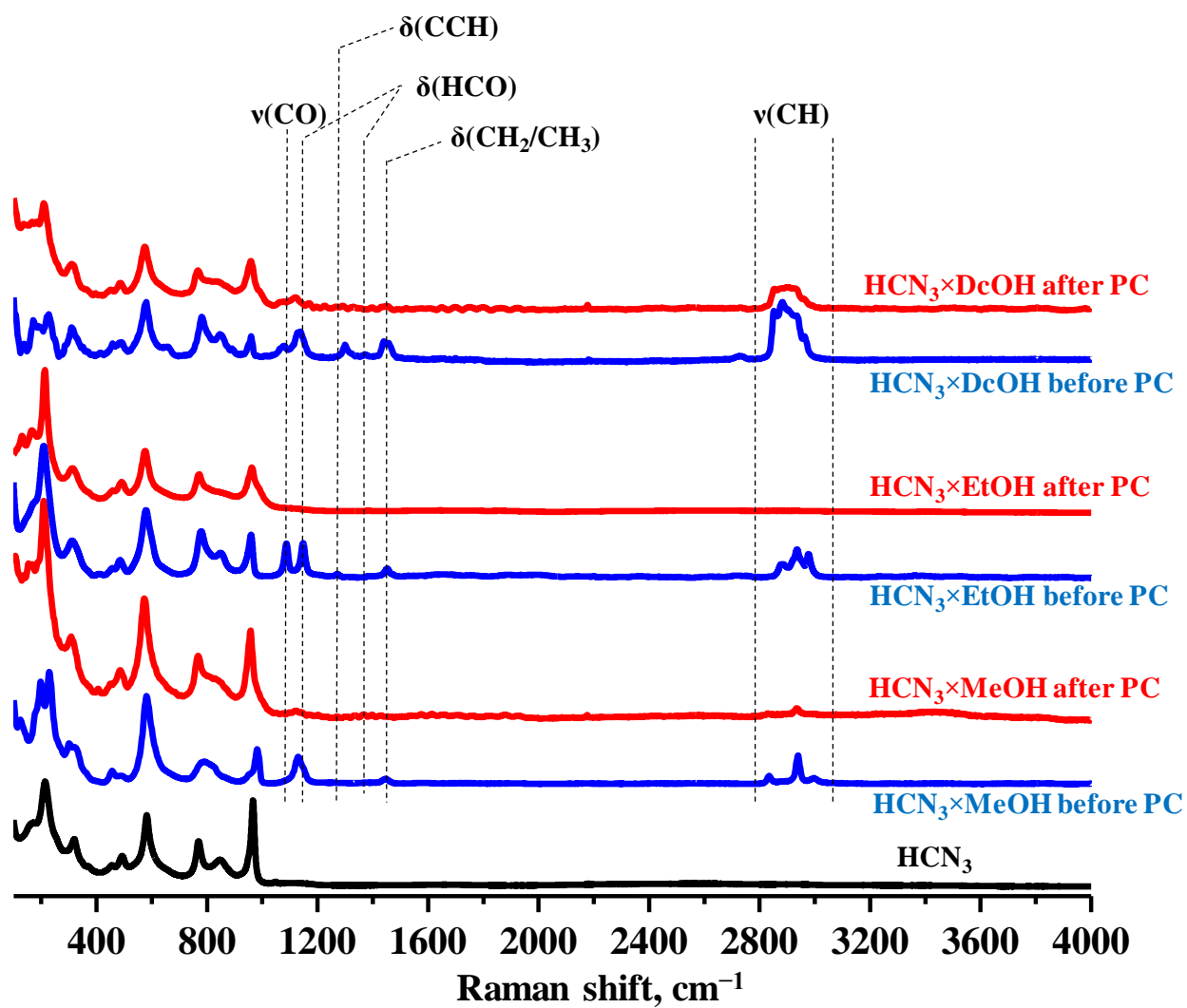
Supporting Information S7

XRD patterns of some *n*-alkoxy derivatives before and after photocatalytic (PC) experiments

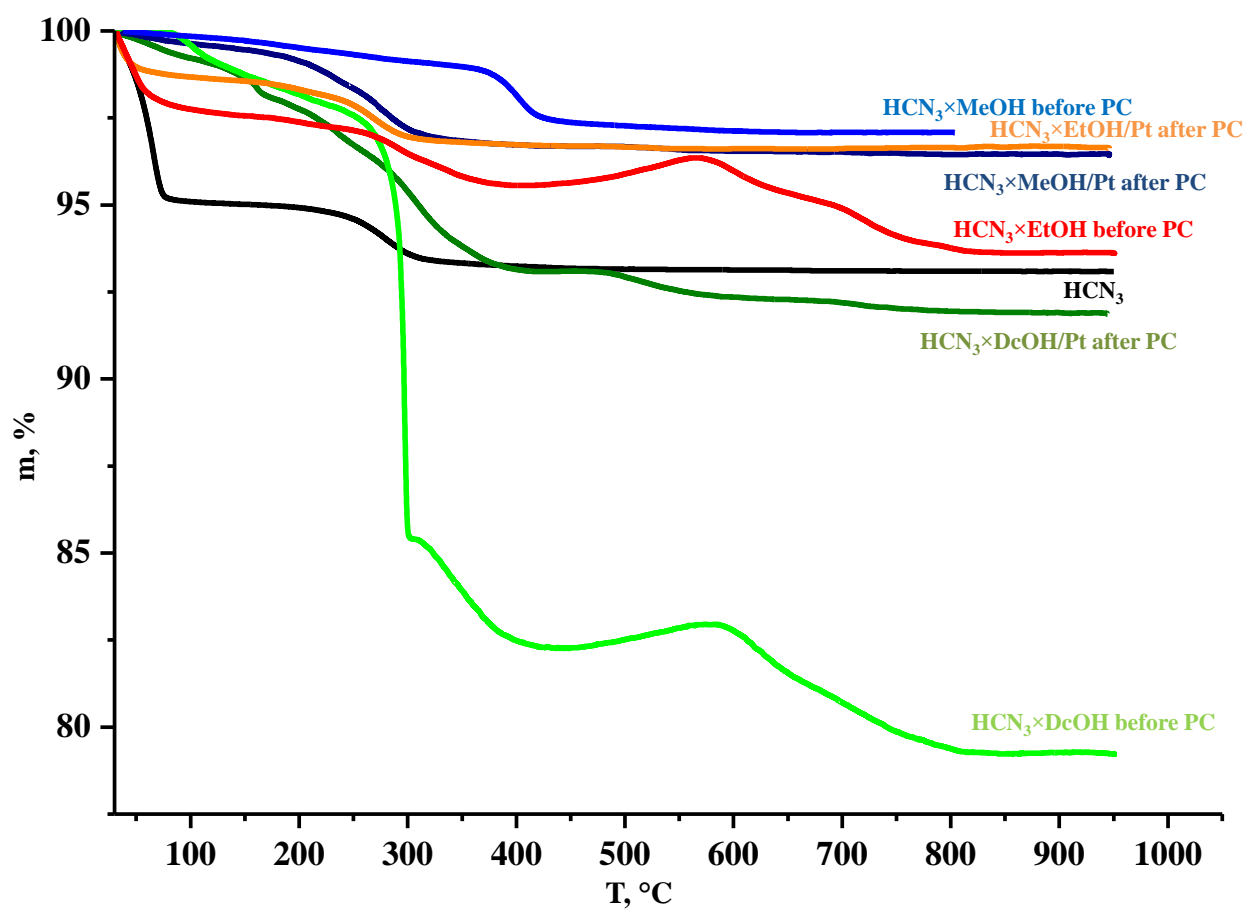Raman spectra of some *n*-alkoxy derivatives before and after photocatalytic (PC) experiments

TG curves of some *n*-alkoxy derivatives before and after photocatalytic (PC) experiments

Supporting Information S10

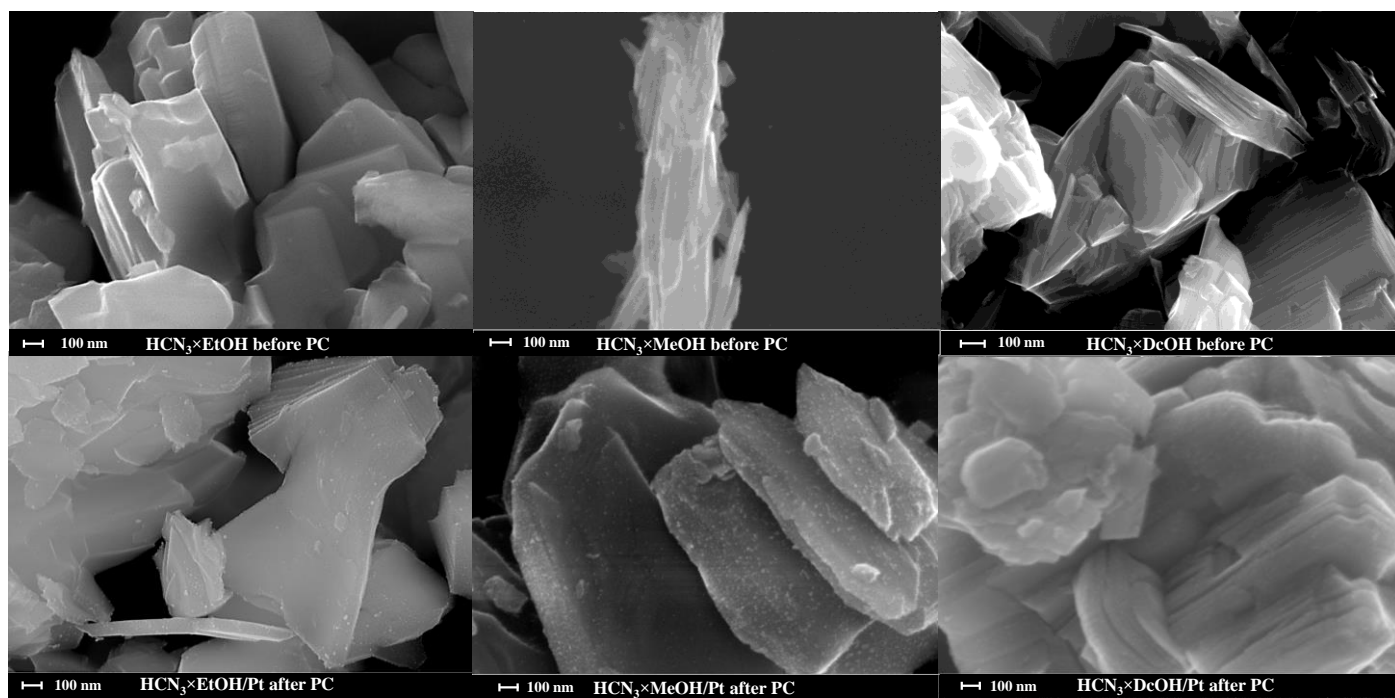SEM images of some *n*-alkoxy derivatives before and after photocatalytic (PC) experiments
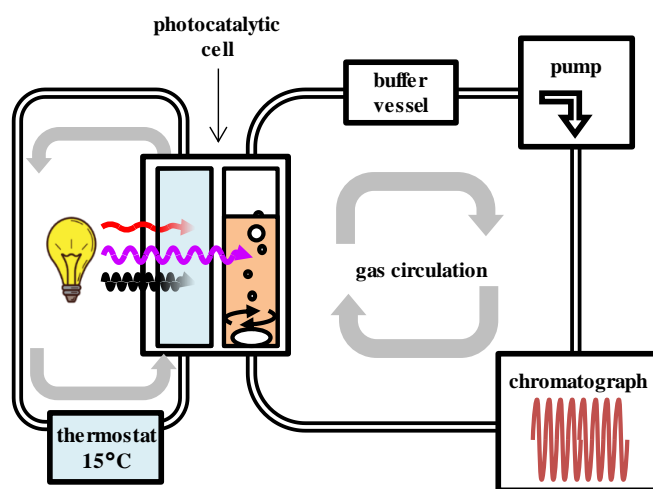

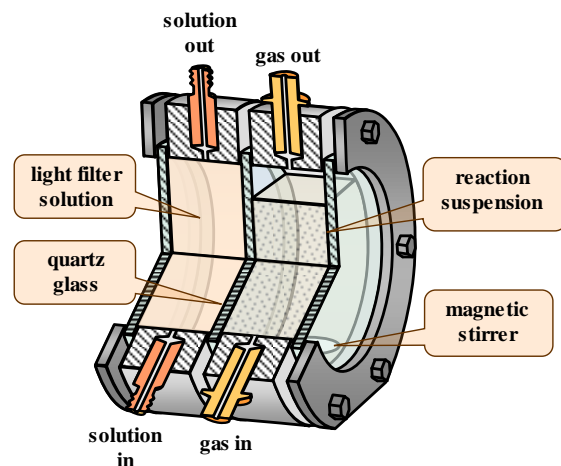|		
100 nm HCN₃×EtOH before PC	100 nm HCN₃×MeOH before PC	100 nm HCN₃×DcOH before PC
100 nm HCN₃×EtOH/Pt after PC	100 nm HCN₃×MeOH/Pt after PC	100 nm HCN₃×DcOH/Pt after PC

Supporting Information S11

Scheme of (a) photocatalytic setting and (b) reaction cell

(a)

(b)

Spectrophotometric calibrations for express measurement of photocatalytic suspensions' concentrations

To prepare the calibration plot for suspensions of the bulk non-exfoliated niobate for determination concentrations c_1 and c_2 during photocatalytic experiments, 30 mg of HCN_3 was added to 60 ml of 1 mol. % methanol. Then the mixture was sonicated for 10 min in an Elmasonic S10H bath (60 W). Afterwards, the suspension obtained was used to build the spectrophotometric calibration dependence in coordinates optical density (A) – niobate concentration in mg/l (c). For this, a series of spectra with various suspension dilutions was recorded, analytical wavelength $\lambda = 550$ nm was selected and the linear approximation of the experimental dependence $A_\lambda = A_\lambda$ (c) was found using the least-squares method (graph a).

To prepare the calibration plot for suspensions of the niobate exfoliated into nanolayers for determination concentration c_3 during photocatalytic experiments, 30 mg of HCN_3 was placed into a glass tube with 30 ml of 0.004 M aqueous tetrabutylammonium hydroxide (TBAOH) and sonicated by a Hielscher UP200St (200 W) homogenizer at half power for 5 min. After shaking at room temperature for 24 h, the suspension was sonicated for 5 min again. Hereafter bulk non-exfoliated particles were separated via centrifuging at a separation factor F = 1000 for 1 h and concentration of the suspension obtained was determined by ICP-AES after preliminary acid digestion. To obtain the calibration plot A – c (mg/l), a series of spectra with various suspension dilutions was recorded, analytical wavelength $\lambda = 230$ nm was selected and the linear approximation of the experimental dependence $A_\lambda = A_\lambda$ (c) was found using the least-squares method (graph b).

(a)

$$A_{550} = 0.0048c - 0.008$$

(b)

$$A_{230} = 0.072c + 0.0034$$