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Compound based on star-shaped oligophenylene and fullerene C_{60}

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Methods.

X-ray photoelectron spectra were acquired with an Axis Ultra DLD (Kratos, UK) spectrometer using monochromatized Al Kα (1486.6 eV) radiation at an operating power of 150 W of the X-ray tube. Survey and high-resolution spectra of appropriate core levels were recorded at pass energies of 160 and 40 eV and with step sizes of 1 and 0.1 eV, respectively. Sample area of 300 μm × 700 μm contributed to the spectra. The samples were mounted on a sample holder with a two-sided adhesive tape, and the spectra were collected at room temperature. The base pressure in the analytical UHV chamber of the spectrometer during measurements did not exceed 10^{-8} Torr. The energy scale of the spectrometer was calibrated to provide the following values for reference samples (i.e., metal surfaces freshly cleaned by ion bombardment): Au $4f_{7/2}$–83.96 eV, Cu $2p_{3/2}$–932.62 eV, Ag $3d_{5/2}$–368.21 eV. The electrostatic charging effects were compensated by using an electron neutralizer. Sample charging was corrected by referencing to the C-C/C-H peak identified in the C ls spectrum (284.8 eV). After charge referencing, a Shirley-type background with inelastic losses was subtracted from the high-resolution spectra. The surface chemical composition was calculated using atomic sensitivity factors included in the software of the spectrometer corrected for the transfer function of the instrument.

Preparation of C_{60} N-monohydrofullerenyl-L-valine methyl ester **2a**

Amino acid fullerene derivative (AFD) **2a** was synthesized as reported [A. Yu. Belik *et al*., *Spectrochimica Acta, Part A, Molecular and Biomolecular Spectroscopy*, 2021, **260**, 119885. https://doi.org/10.1016/j.saa.2021.119885]. An aqueous solution of L-valine potassium salt (0.3169 g, 2.07 mmol) and 18-crown-6 (0.5465 g, 2.07 mmol) were added to a solution of fullerene C_{60} (0.3 g, 0.414 mmol) in *o*-dichlorobenzene (10 ml). The reaction mixture was stirred at 60 °C for 6-8 hours. The solvent was distilled off, the residue was dissolved in water, acidified, and treated with saturated KCl solution. The precipitate was filtered off, washed with water, and dried. The yield was quantitative. The obtained *N*-(monohydrofullerenyl)valine (acid

form Val-C_{60}) was soluble in DMF, DMSO and pyridine. To obtain methyl ester Val-C_{60} **2a**, potassium salt Val-C_{60} **2b** was used. To a solution of acid form Val-C_{60} (0.3465 g, 0.414 mmol) in pyridine (5 ml), a solution of KOH (0.0464 g, 0.828 mmol) in 1 ml of water was added to form salt **2b**. Then the solution was stirred at room temperature for ca. 30 min and put on dialysis against water. To the resulting aqueous solution, iodomethane (0.3 g, 2.07 mmol) was added, and this was boiled for 6 hours. The precipitate of (Val-C_{60}) methyl ester **2a** was filtered off. The yield was quantitative.

Preparation of **3**

To a solution of compound **2a** (104 mg, 0.12 mmol) of in pyridine (5 ml) was added compound **1** (32.4 mg, 0.04 mmol), this boiled for 8 h under argon and left overnight at room temperature. Then, ethylene chlorohydrin (0.5 ml) was added, and this was stirred at room temperature for 4 h. The resulting mixture was placed on dialysis against water. The precipitate that formed was centrifuged, dried and extracted in chloroform. The yield of **3** was almost quantitative.

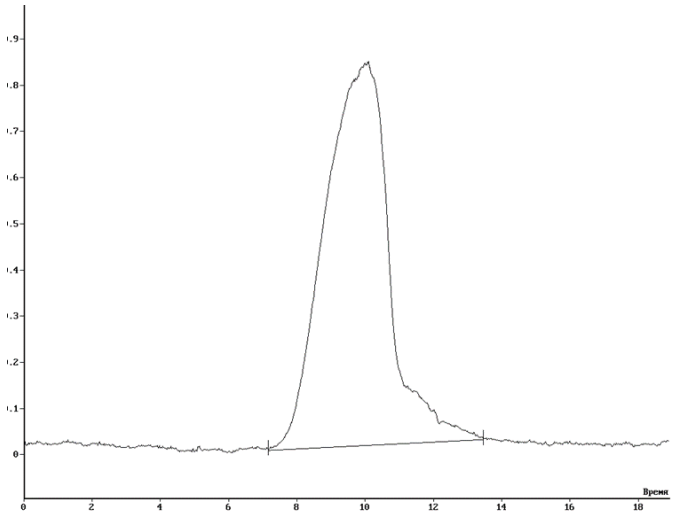

Figure S1 GPC of **3.** According to the retention time: M = 3081, M_n = 4383; M_w = 8263; D = 1.885

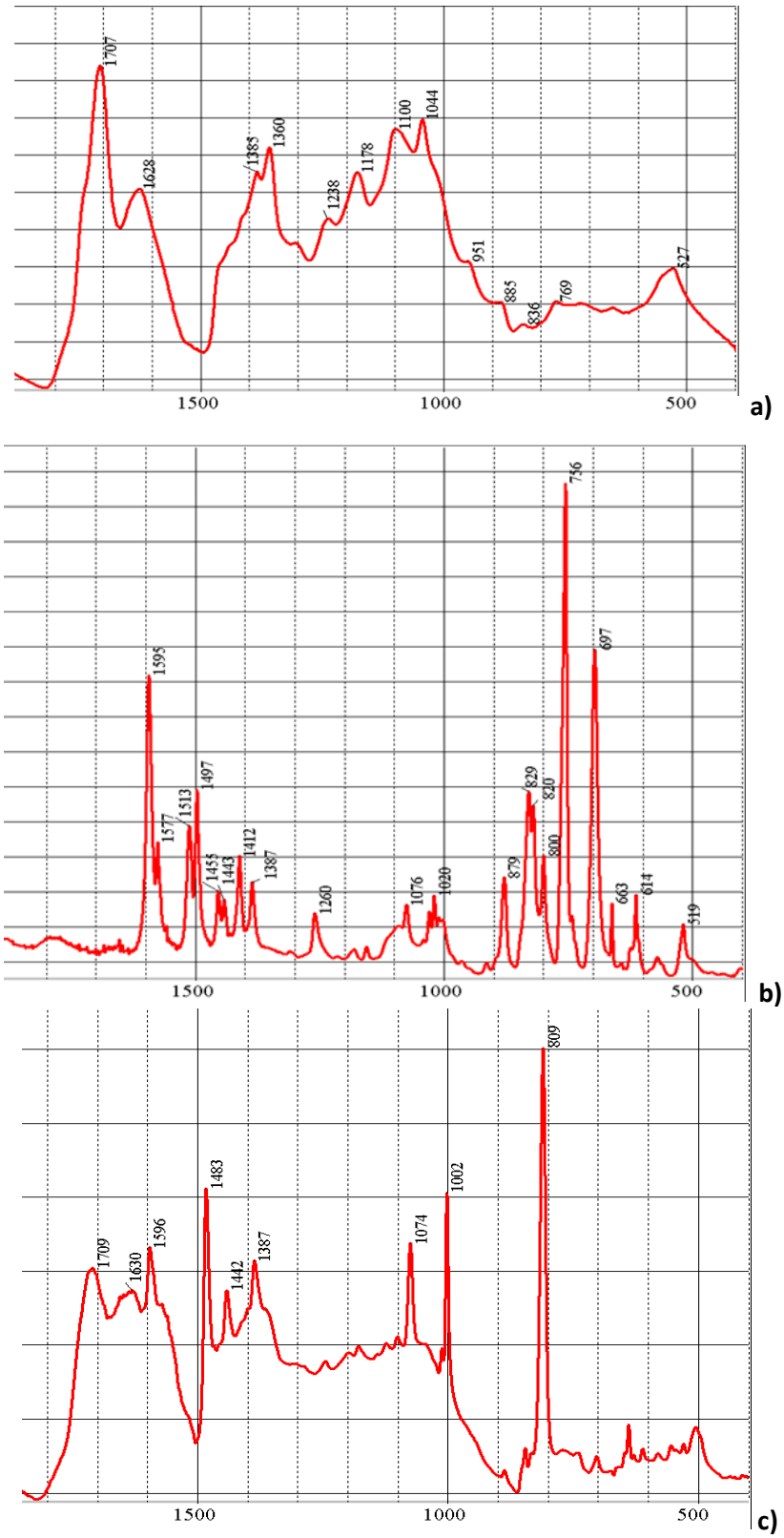

Figure S2 IR spectra of **3** (a), **1** (b) and **4** (c).

Table S1 Characteristics of the photoelectron spectra: binding energies (E_b), Gaussian widths (W), and relative intensities (I_{rel}) of photoelectron peaks belonging to different chemical groups in the C 1s and N 1s spectra.

Cmpd.	Group	C-C/ C-H	C-N	C-O-C/C-OH	C=O/O-C-O	**C**(O)O	Sat1	Sat2	Sat3	Sat4
C_{60}	E_b, eV	284.8					286.5	288.7	289.8	290.8
	W, eV	0.68					0.9	0.86	1.00	1.2
	I_{rel}	0.87					0.04	0.03	0.02	0.04
4	E_b, eV	284.7		285.4			289.0	290.5	291.5	292.6
	W, eV	0.8		0.81			1.1	1.1	1.1	1.2
	I_{rel}	0.82		0.11			0.02	0.02	0.02	0.01
2a	E_b, eV	284.8	285.7	286.5	287.5	289.2				
	W, eV	1.06	0.95	1.1	1.2	1.3				
	I_{rel}	0.60	0.07	0.22	0.09	0.03				
3	E_b, eV	284.8	285.9	286.5	287.8	289.0				
	W, eV	1.17	1.2	1.2	1.2	1.2				
	I_{rel}	0.66	0.06	0.16	0.07	0.05				

The survey XPS spectra of the samples studied are displayed in Figs. S3-S6.

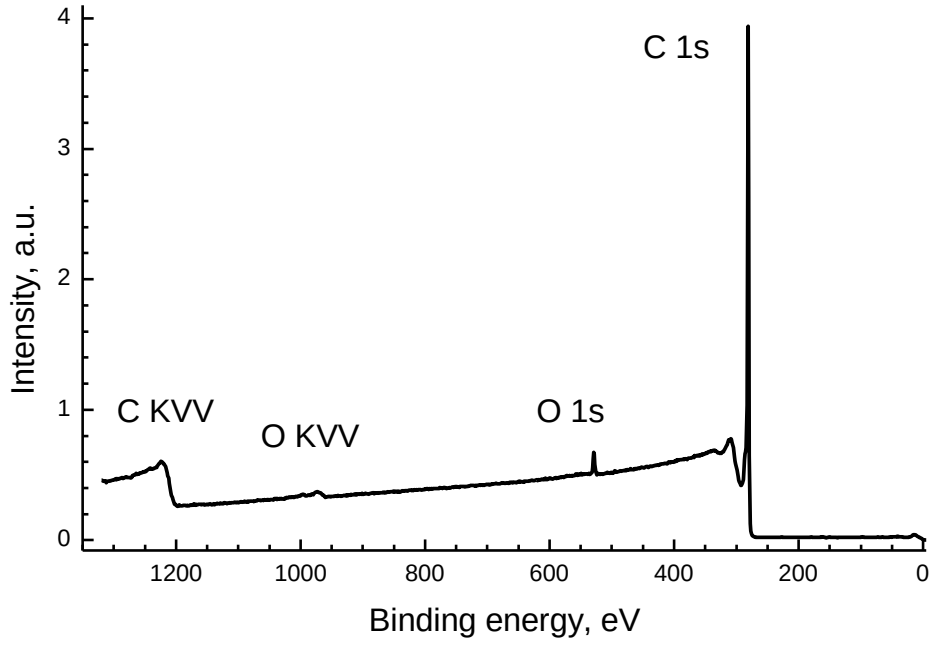

Figure S3 The survey spectrum of sample **1**.

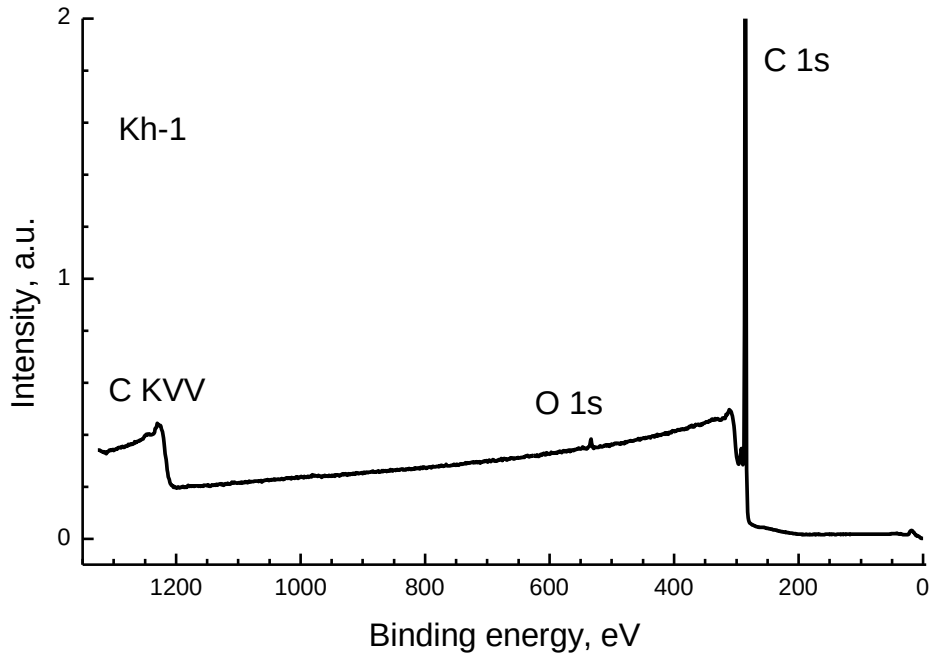

Figure S4 The survey spectrum of sample **4**.

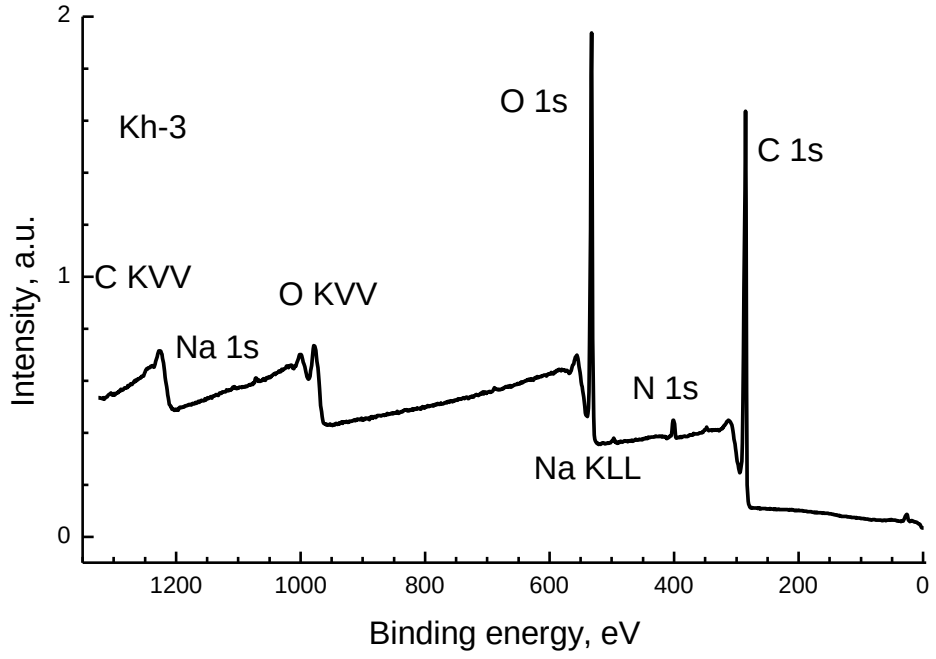

Figure S5 The survey spectrum of sample potassium salt **2b**.

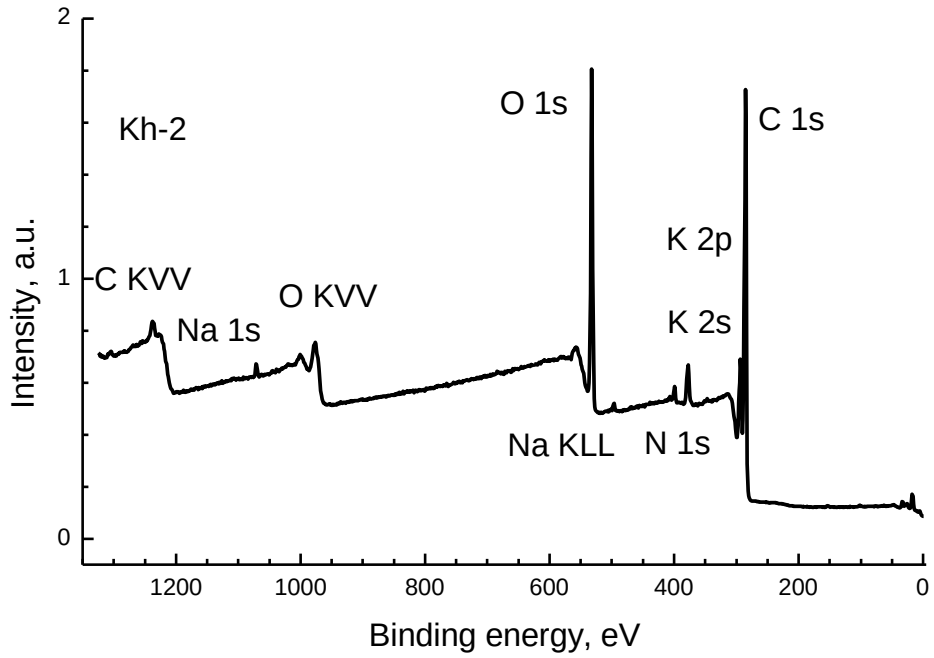

Figure S6 The survey spectrum of sample **3**.