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Synthesis of Novel Functionally Substituted Esters of *m*-Carborane-1,7-dicarboxylic Acid

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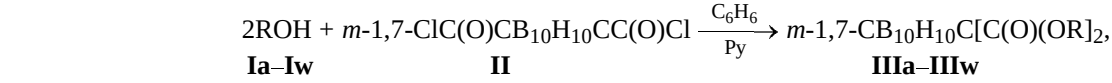
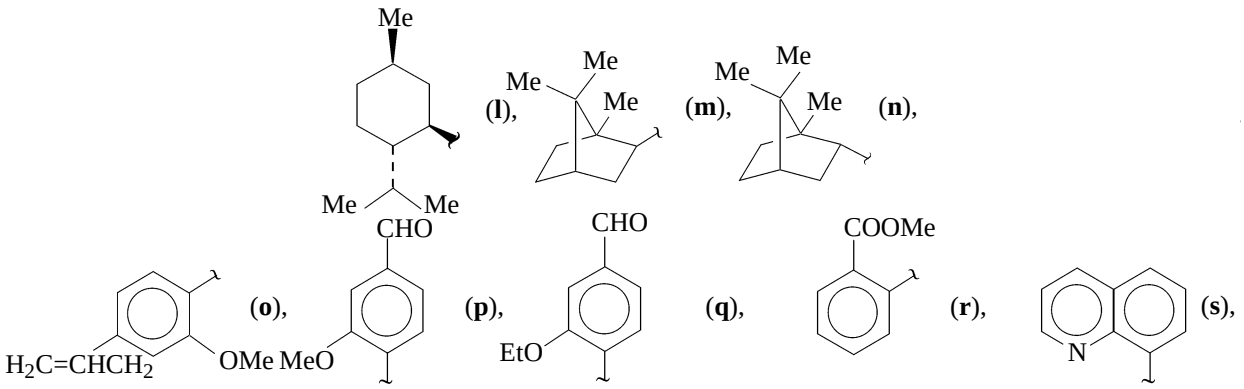
Abstract — *m*-Carborane-1,7-dicarbonyl dichloride was reacted with functionally substituted alcohols and phenols to obtain previously unknown esters, peroxy-containing inclusive.

One of the perspective lines of the search for agents for diagnostics and treatment of cancer is synthesis of functionally substituted carborane derivatives [1–7]. In the present work we describe the synthesis of novel functionally substituted *m*-carborane-1,7-dicarboxylic acid esters **IIIa**–**IIIw**. As starting compounds we used fuctionally substituted alcohols and phenols **Ia**–**Iw**, including terpene **Ib**, **Ic**, and **Il**–**I**, steroid **It**–**Iw**, vegetable **Io**–**Iq**, peroxy-containing **Ih**–**Ik**, and acetylenic **Id**–**If** and **Ih**–**Ik** [8–11]. Esters **IIIa**–**IIIw** were obtained by refluxing *m*-carborane-1,7-dicarbonyl dichloride (**II**) with compounds **Ia**–**Iw** in absolute benzene in the presence of pyridine. The yields of the target products were 71–90%.

The structure of esters **IIIa**–**IIIw** was confirmed by their elemental analyses, ^{1}H NMR, IR, and UV spectra. The yields and physicochemical characteristics of compounds **IIIa**–**IIIw** are listed in Table 1, the ^{1}H NMR spectra, in Table 2, and IR and UV spectra, in Table 3. The purity of the esters, as judged from the ^{1}H NMR spectra, was 98±1%.



I, III, R = $Me(CH_2)_{16}$ (**a**), $Me_2C{=}CH(CH_2)_2CHMe(CH_2)_2$ (**b**), $Me_2C{=}CH(CH_2)_2CMe{=}CHCH_2$ (**c**), $HC{\equiv}C(CH_2)_2$ (**d**), $HC{\equiv}C(CH_2)_3$ (**e**), HC≡CCMe2 (**f**), $MeC(O)CH_2$ (**g**), $EtMe_2COOCMe_2C{\equiv}CCH(CH_2)_2Me$ (**h**), $Me_3COOCMe_2C{\equiv}CCH\cdot(CH_2)_6Me$ (**i**), $EtMe_2COOCMe_2C{\equiv}CCH(CH_2)_6Me$ (**j**), *p*-$PrMe_2COOCMe_2C{\equiv}CCH(CH_2)_6Me$ (**k**),



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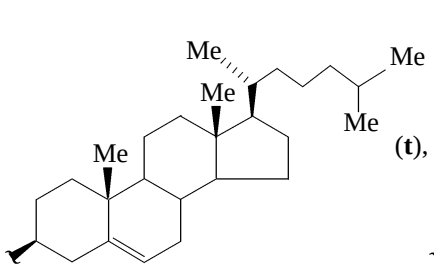

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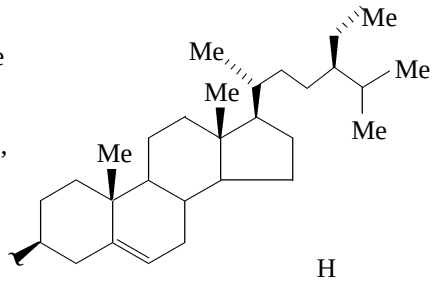

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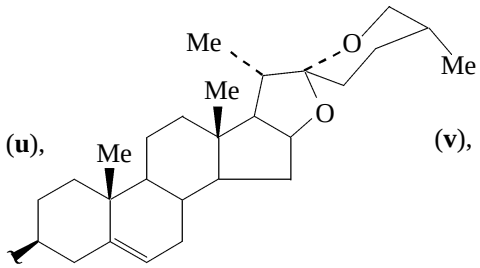
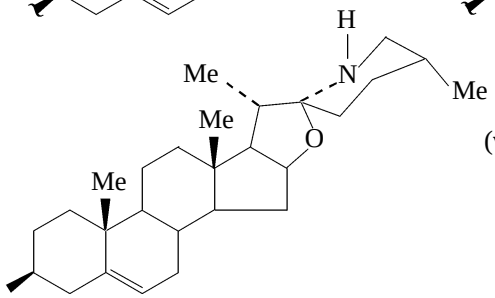


(**v**),



(**w**).

Table 1. Properties of esters **IIIa**–**IIIw**

Comp. no.	Yield, %	mp, °C	d_4^{20}	n_D^{20}	Found, %			Formula	Calculated, %			*M*	
					C	H	B		C	H	B	found	calculated
IIIa	79	31–32	–	–	64.51	11.48	15.09	$C_{38}H_{80}B_{10}O_4$	64.36	11.37	15.25	698.3	709.2
IIIb	83	–	0.9952	1.5020	57.01	10.03	21.10	$C_{24}H_{48}B_{10}O_4$	56.66	9.51	21.25	497.2	508.8
IIIc	80	–	1.0431	1.5150	57.42	9.02	21.13	$C_{24}H_{44}B_{10}O_4$	57.11	8.79	21.42	494.6	504.7
IIId	88	–	1.0330	1.5220	43.04	6.12	31.87	$C_{12}H_{20}B_{10}O_4$	42.85	5.99	32.14	322.7	336.4
IIIe	90	–	1.0602	1.5200	46.36	6.81	29.30	$C_{14}H_{24}B_{10}O_4$	46.14	6.64	29.66	349.0	364.5
IIIf	75	38–39	–	–	46.29	6.72	29.49	$C_{14}H_{24}B_{10}O_4$	46.14	6.64	29.66	351.5	364.5
IIIg	77	–	1.2800	1.5205	35.07	6.04	31.04	$C_{10}H_{20}B_{10}O_6$	34.88	5.85	31.39	328.1	344.4
IIIh	80	–	1.0242	1.4865	56.69	9.03	15.65	$C_{32}H_{60}B_{10}O_8$	56.44	8.88	15.88	661.4	680.9
IIIi	85	–	0.9895	1.4795	59.83	9.67	13.92	$C_{38}H_{72}B_{10}O_8$	59.65	9.48	14.13	747.8	765.1
IIIj	81	–	0.9978	1.4840	60.74	9.81	13.44	$C_{40}H_{76}B_{10}O_8$	60.57	9.66	13.63	778.5	793.2
IIIk	80	–	1.0255	1.4825	61.76	10.04	12.89	$C_{42}H_{80}B_{10}O_8$	61.43	9.82	13.17	804.9	821.2
IIIl	76	–	1.0113	1.5060	56.80	9.64	21.04	$C_{24}H_{48}B_{10}O_4$	56.66	9.51	21.25	495.8	508.8
IIIm	78	132–133	–	–	57.32	8.94	21.30	$C_{24}H_{44}B_{10}O_4$	57.11	8.79	21.42	492.5	504.7
IIIn	75	104–105	–	–	57.35	8.89	21.33	$C_{24}H_{44}B_{10}O_4$	57.11	8.79	21.42	495.0	504.7
IIIo	84	67–68	–	–	55.12	6.31	20.37	$C_{24}H_{32}B_{10}O_6$	54.95	6.15	20.61	507.3	524.6
IIIp	88	33–34	–	–	48.23	5.05	21.38	$C_{20}H_{24}B_{10}O_8$	47.99	4.83	21.60	490.4	500.5
IIIq	87	75–76	–	–	50.19	5.46	20.33	$C_{22}H_{28}B_{10}O_8$	49.99	5.34	20.45	509.7	528.6
IIIr	83	103–104	–	–	48.11	5.00	21.41	$C_{20}H_{24}B_{10}O_8$	47.99	4.83	21.60	491.2	500.5
IIIs[a]	83	222–223	–	–	54.43	4.66	22.09	$C_{22}H_{22}B_{10}N_2O_4$	54.31	4.56	22.22	470.1	486.5
IIIt	76	233–234	–	–	72.04	10.63	10.95	$C_{58}H_{100}B_{10}O_4$	71.85	10.40	11.15	931.8	969.5
IIIu	71	223–224	–	–	72.77	10.74	10.30	$C_{62}H_{108}B_{10}O_4$	72.61	10.61	10.54	993.5	1025.6
IIIv	80	275–276	–	–	68.19	9.21	10.22	$C_{58}H_{92}B_{10}O_8$	67.93	9.04	10.54	989.7	1025.5
IIIw[b]	75	315 (dec.)	–	–	68.21	9.40	10.35	$C_{58}H_{94}B_{10}N_2O_6$	68.06	9.26	10.56	987.1	1023.5

[a] Found N, %: 5.61. Calculated N, %: 5.76. [b] Found N, %: 2.58. Calculated N, %: 2.74.

Table 2. ^{1}H NMR spectra of esters **IIIa**–**IIIw**

Comp. no.	δ, ppm
IIIa	–1.00–6.00 m (10H, $B_{10}H_{10}$), 0.89 t (6H, 2Me, ^3J 6.0 Hz), 1.10–1.85 m [60H, $2(CH_2)_{15}$], 4.14 t (4H, $2CH_2O$, ^3J 6.0 Hz)
IIIb	–1.00–6.00 m (10H, $B_{10}H_{10}$), 0.91 d (6H, $2C^3Me$, ^3J 5.7 Hz), 1.00–2.20 m (14H, $2C^3H$, $2C_2H_2$, $2C^4H_2$, and $2C^5H_2$), 1.61 s and 1.69 s (2H, $2C^7Me_2$), 4.18 t (4H, $2C^1H_2$, ^3J 6.5 Hz), 4.85–5.20 m (2H, $2C^6H$)
IIIc	–1.00–6.00 m (10H, $B_{10}H_{10}$), 1.61 s (6H, $2C^3Me$), 1.69 s (2H, $2C^7Me_2$), 0.80–2.15 m (8H, $2C^4H_2$, and $2C^5H_2$), 4.63 d (4H, $2C^1H_2$, ^3J 6.8 Hz), 4.85–5.35 m (4H, $2C^2H$ and $2C^6H$)
IIId	–1.00–6.00 m (10H, $B_{10}H_{10}$), 2.00 t (2H, $2C{\equiv}CH$, ^4J 2.6 Hz), 2.54 t.d [4H, $^3J(CH_2)$ 6.8, $2CH_2C{\equiv}C$, $^4J(C{\equiv}CH)$ 2.6 Hz], 4.23 t (4H, $2CH_2O$, ^3J 6.8 Hz)
IIIe	–1.00–6.00 m (10H, $B_{10}H_{10}$), 1.70–2.40 m [10H, $2C{\equiv}CH$, and $2(CH_2)_2C{\equiv}C$], 4.25 t (4H, $2CH_2O$, ^3J 6.3 Hz)
IIIf	–1.50–6.00 m (10H, $B_{10}H_{10}$), 1.67 s (12H, $2Me_2C$), 2.58 s (2H, $2C{\equiv}CH$)
IIIg	–1.00–6.00 m (10H, $B_{10}H_{10}$), 2.15 s (6H, 2Me), 4.69 s (4H, $2CH_2$)
IIIh	–1.00–6.00 m (10H, $B_{10}H_{10}$), 0.75–1.05 m (12H, 4Me), 1.20 s (12H, $2Me_2COO$), 1.45 s (12H, $2Me_2C$), 1.30–2.00 m [12H, $2CH_2$ and $2(CH_2)_2$], 5.30 t (2H, 2CHO, ^3J 7.3 Hz)
IIIi	–1.00–6.00 m (10H, $B_{10}H_{10}$), 0.75–1.00 m (6H, 2Me), 1.22 s (18H, $2Me_3COO$), 1.15–1.90 m [24H, $2(CH_2)_6$], 1.45 s (12H, $2Me_2C$), 5.25 t (2H, 2CHO, ^3J 7.1 Hz)
IIIj	–1.00–6.00 m (10H, $B_{10}H_{10}$), 0.75–1.05 m (12H, 4Me), 1.10–1.90 m [28H, $2CH_2$, and $2(CH_2)_6$], 1.20 s (12H, $2Me_2COO$), 1.45 s (12H, $2Me_2C$), 5.27 t (2H, 2CHO, ^3J 7.2 Hz)
IIIk	–1.00–6.00 m (10H, $B_{10}H_{10}$), 0.75–1.05 m (12H, 4Me), 1.15–1.95 m [32H, $2(CH_2)_2$, and $2(CH_2)_6$], 1.21 s (12H, $2Me_2COO$), 1.45 s (12H, $2Me_2C$), 5.30 t (2H, 2CHO, ^3J 7.2 Hz)
IIIl	–1.00–6.00 m (10H, $B_{10}H_{10}$), 0.74 d (6H, $2C^1Me$, ^3J 6.7 Hz), 0.91 d (12H, $2C^8Me_2$, ^3J 6.7 Hz), 0.85–2.05 m (18H, $2C^1H$, $2C^2H_2$, $2C^4H$, $2C^5H_2$, $2C^6H_2$ and $2C^8H$), 4.45–4.80 m (2H, $2C^3H$)
IIIm	–1.00–6.8 0 m (10H, $B_{10}H_{10}$), 0.85 s (6H, $2Me^{10}$), 0.92 s (12H, $2Me^8$, and $2Me^9$), 0.95–2.50 m (14H, $2C^3H_2$, $2C^4H$, $2C^5H_2$ and $2C^6H_2$), 4.75–4.95 m (2H, $2C^2H_{exo}$)
IIIn	–1.00–6.20 m (10H, $B_{10}H_{10}$), 0.85 s (12H, $2Me^9$, and $2Me^{10}$), 0.98 s (6H, $2Me^8$), 0.75–2.20 m (14H, $2C^3H_2$, $2C^4H$, $2C^5H_2$ and $2C^6H_2$), 4.50–4.75 m (2H, $2C^2H_{endo}$)
IIIo	–1.00–6.00 m (10H, $B_{10}H_{10}$), 3.36 d (4H, $2CH_2O$, ^3J 6.7 Hz), 3.79 s (6H, 2Me), 4.92–5.25 m (4H, $2{=}CH_2$), 5.67–6.23 m (2H, 2=CH), 6.55–6.97 m (6H, $2C_6H_3$)
IIIp	–1.00–6.00 m (10H, $B_{10}H_{10}$), 3.90 s (6H, 2Me), 7.12–7.55 m (6H, $2C_6H_3$), 9.93 s (2H, 2CHO)
IIIq	–1.00–6.00 m (10H, $B_{10}H_{10}$), 1.43 t (6H, 2Me, ^3J 7.4 Hz), 4.11 q (4H, $2CH_2$, ^3J 7.4 Hz), 7.10–7.50 m (6H, $2C_6H_3$), 9.92 s (2H, 2CHO)
IIIr	–1.00–6.00 m (10H, $B_{10}H_{10}$), 3.90 s (6H, 2Me), 6.90–8.10 m (8H, $2C_6H_4$)
IIIs	–1.00–6.00 m (10H, $B_{10}H_{10}$), 7.20–8.90 m (12H, $2C_9H_6N$)
IIIt	–1.00–6.00 m (10H, $B_{10}H_{10}$), 0.69 s (6H, $2Me^{18}$), 0.80–1.00 m (18H, $2Me^{21}$, $2Me^{26}$, and $2Me^{27}$), 1.03 s (6H, $2Me^{19}$), 0.80–2.45 m (56H, CH_2 and CH), 4.35–4.75 m (2H, $2C^3H$), 5.27–5.47 m (2H, $2C^6H$)
IIIu	–1.00–6.00 m (10H, $B_{10}H_{10}$), 0.69 s (6H, $2Me^{18}$), 1.02 s (6H, $2Me^{19}$), 0.65–2.45 m (82H, 8Me, CH_2, and CH), 4.30–4.85 m (2H, $2C^3H$), 5.35–5.50 m (2H, $2C^6H$)
IIIv	–1.00–6.00 m (10H, $B_{10}H_{10}$), 0.80 s (6H, $2Me^{18}$), 1.05 s (6H, $2Me^{19}$), 0.70–2.45 m (60H, 4Me, CH_2, and CH), 3.20–4.75 m (8H, $2C^3H$, $2C^{16}H$ and $2CH_2O$), 5.35–5.50 m (2H, $2C^6H$)
IIIw	–1.00–6.00 m (10H, $B_{10}H_{10}$), 0.82 s (6H, $2Me^{18}$), 1.04 s (6H, $2Me^{19}$), 0.80–1.15 m (12H, 2Me), 1.15–3.00 m (52H, CH_2, and CH), 3.20–4.80 m (4H, $2C^3H$ and $2C^{16}H$), 5.20–5.50 m (2H, $2C^6H$)

EXPERIMENTAL

The IR spectra were obtained on a Protege-460 Fourier spectrometer in KBr pellets (**IIIa, IIIf, IIIm**–**IIIw**) or in thin films (**IIIb**–**IIIe**, **IIIg**–**IIIl**). The UV spectra were measured on a Specord UV-Vis instrument for 1×10^{-3} M solutions in 1-butanol. The ^{1}H NMR spectra were run on a Tesla-567A spectrometer (100 MHz) in $CDCl_3$ against internal TMC. The molecular weights were measured by cryoscopy in benzene. The starting *m*-carborane-1,7-dicarbonyl dichloride (**II**) was prepared by the procedure in [1],

Table 3. IR and UV spectra of esters **IIIa**–**IIIw**

Comp. no.	IR spectrum, ν, cm^{-1}	UV spectrum, λ_{max}, nm (ε)
IIIa	2955, 2918, 2850 (CH_{Alk}); 2621, 2598, 2586 (BH); 1743 (C=O); 1471 (CH_2); 1263 (C–O); 740, 719 (BH)	201 (100), 211 (150), 220 (100)
IIIb	3030 (=CH); 2964, 2927, 2915, 2873, 2855 (CH_{Alk}); 2619 (BH); 1749 (C=O); 1670 (C=C); 1458 (CH_2); 1260 (C–O); 760, 737 (BH)	204 (8000)
IIIc	3055, 3025 (=CH); 2966, 2927, 2875, 2856 (CH_{Alk}); 2618 (BH); 1747 (C=O); 1660, 1643 (C=C); 1452 (CH_2); 1256 (C–O); 760, 737 (BH)	206 (16000)
IIId	3304 (≡CH); 2970, 2915, 2900, 2850 (CH_{Alk}); 2619 (BH); 2130 (C≡C); 1748 (C=O); 1459 (CH_2); 1260 (C–O); 764, 736 (BH); 649 (≡CH)	201 (100), 208 (500), 220 (400)
IIIe	3304 (≡CH); 2965, 2935, 2900, 2850 (CH_{Alk}); 2619 (BH); 2120 (C≡C); 1748 (C=O); 1460, 1434 (CH_2); 1259 (C–O); 765, 735 (BH); 642 (≡CH)	201 (100), 208 (500), 221 (400)
IIIf	3288, 3270 (≡CH); 3010, 2992, 2945, 2925 (CH_{Alk}); 2950, 2625, 2606, 2595, 2580 (BH); 2125 (C≡C); 1752 (C=O); 1285, 1267, 1122 (C–O); 740 (BH); 625 (≡CH)	202 (100), 208 (500), 220 (400)
IIIg	2997, 2941 (CH_{Alk}); 2621 (BH); 1759, 1736 (C=O); 1421 (CH_2); 1276, 1259, 1181, 1139, 1034 (C–O); 754, 735 (BH)	210 (500), 220 (100), 255 (200)
IIIh	2968, 2937, 2877 (CH_{Alk}); 2619 (BH); 1752 (C=O); 1463 (CH_2); 1243, 1160, 1118, 1012, 928 (C–O); 870 (O–O); 760, 740 (BH)	203 (100), 208 (500), 220 (400)
IIIi	2980, 2957, 2930, 2859 (CH_{Alk}); 2618 (BH); 1751 (C=O); 1466 (CH_2); 1250, 1161, 1120, 1011, 946 (C–O); 870 (O–O); 760, 740 (BH)	203 (100), 207 (500), 221 (400)
IIIj	2980, 2960, 2930, 2858 (CH_{Alk}); 2619 (BH); 1752 (C=O); 1463 (CH_2); 1250, 1159, 1120, 1010, 946 (C–O); 859 (O–O); 760, 736 (BH)	202 (100), 208 (500), 220 (400)
IIIk	2983, 2958, 2931, 2872, 2859 (CH_{Alk}); 2618 (BH); 1752 (C=O); 1465 (CH_2); 1250, 1159, 1120, 1010, 958 (C–O); 875 (O–O); 765, 740 (BH)	203 (100), 208 (500), 220 (400)
IIIl	2958, 2927, 2871 (CH_{Alk}); 2618 (BH); 1743 (C=O); 1456 (CH_2); 1257, 1013, 957 (C–O); 756, 736 (BH)	201 (100), 211 (150), 220 (100)
IIIm	2980, 2956, 2884, 2865 (CH_{Alk}); 2622, 2598, 2570 (BH); 1740 (C=O); 1474, 1454 (CH_2); 1262, 1016, 974 (C–O); 754, 736 (BH)	201 (100), 211 (150), 220 (100)
IIIn	3002, 2981, 2956, 2877 (CH_{Alk}); 2619 (BH); 1743 (C=O); 1477, 1456 (CH_2); 1262, 1047, 1013, 964 (C–O); 760, 740 (BH)	201 (100), 210 (150), 221 (100)
IIIo	3079 (=CH and CH_{Ar}); 2997, 2977, 2939, 2916, 2850, 2835 (CH_{Alk}); 2670, 2619, 2600, 2579 (BH); 1766 (C=O); 1640 (C=C); 1606, 1513, 1465 (Ar); 1450, 1424 (CH_2); 1270, 1250, 1240, 1195, 1184, 1149, 1123, 1035, 1000 (C–O); 913, 845, 825, 793 (CH_{Ar}); 753, 735 (BH)	207 (28000), 218 (16000), 272 (6000), 281 (5000)
IIIp	3120, 3075, 3015 (CH_{Ar}); 2970, 2941, 2855, 2835 (CH_{Alk}); 2740 (CHCHO); 2618 (BH); 1771, 1702 (C=O); 1605, 1503, 1465, 1423, 1390 (Ar); 1279, 1237, 1194, 1144, 1119, 1029, 1002 (C–O); 850, 831, 797, 778, 732 (CH_{Ar}); 760, 740 (BH)	207 (17000), 210 (16000), 222 (15000), 260 (13000), 310 (6000)
IIIq	315, 3070, 3045, 3015 (CH_{Ar}); 2981, 2940, 2895, 2880, 2870, 2850, 2835, 2810 (CH_{Alk}); 2745 (CHCHO); 2655, 2624, 2615, 2600 (BH); 1769, 1698 (C=O); 1601, 1503, 1437, 1391 (Ar); 1470 (CH_2); 1278, 1259, 1238, 1189, 1150, 1118, 1031, 1005 (C–O); 860, 830, 790, 743 (CH_{Ar}); 755, 740 (BH)	206 (18000), 210 (16000), 222 (15000), 258 (14000), 311 (6000)
IIIr	3082, 3024, 2993 (CH_{Ar}); 2948, 2900, 2839 (CH_{Alk}); 2673, 2645, 2620, 2608, 2597, 2575 (BH); 1766, 1728 (C=O); 1609, 1485, 1450, 1432 (Ar); 1273, 1249, 1239, 1203, 1188, 1083, 1006 (C–O); 810, 742, 689 (CH_{Ar} and BH)	205 (30000), 226 (20000), 275 (4000)
IIIs	3070, 3054, 3020 (CH_{Ar}); 2660, 2623, 2575 (BH); 1762 (C=O); 1596, 1572, 1499, 1470, 1427, 1389, 1367 (Ar); 1247, 1226, 1166, 1124, 1073, 1051, 1025, 1001 (C–O); 832, 810, 801, 777 (CH_{Ar}); 761, 735 (BH)	205 (30000), 228 (35000), 230 (35000), 280 (5000), 313 (4000)
IIIt	3040 (=CH); 2940, 2910, 2867, 2855, 2822 (CH_{Alk}); 2636, 2621, 2607 (BH); 1741 (C=O); 1670 (C=C); 1468, 1442 (CH_2); 1271, 1252, 1008, 994 (C–O); 764, 735 (BH)	204 (8000)

Table 3. (Contd.)

Comp. no.	IR spectrum, ν, cm^{-1}	UV spectrum, λ_{max}, nm (ε)
IIIu	3035 (=CH); 2960, 2936, 2900, 2867, 2851 (CH_{Alk}); 2616 (BH); 1743 (C=O); 1664 (C=C); 1466 (CH_2); 1270, 1256, 1133, 1055, 1022, 1008, 958 (C–O); 765, 738 (BH)	204 (8000)
IIIv	3040 (=CH); 2970, 2950, 2928, 2906, 2872, 2850 (CH_{Alk}); 2666, 2617, 2575, 2569 (BH); 1742 (C=O); 1670 (C=C); 1465, 1454 (CH_2); 1271, 1257, 1170, 1128, 1096, 1053, 1007, 982 (C–O); 768, 733 (BH)	204 (8000)
IIIw	3030 (=CH); 2947, 2905, 2871, 2850 (CH_{Alk}); 2610, 2563 (BH); 1739 (C=O); 1670 (C=C); 1459 (CH_2); 1267, 1255, 1136, 1013, 983 (C–O); 754, 737 (BH)	204 (8000)

and the synthesis of compounds **Ih**–**Ik** has been described in [10].

Functionally substituted *m*-carborane-1,7-dicarboxylatic acid esters IIIa–IIIw *(general procedure)*. Compounds **Ia**–**Iw** (10 mmol) were dissolved in 130 ml of absolute benzene. From the resulting solution, about 30 ml of the solvent was azeotropically distilled to remove traces of water adsorbed by starting compounds **Ia**–**Iw**. After cooling, 1.4 g of chloride **II** and 1.2 of absolute pyridine were added in one portion. The mixture was refluxed for 8 h and cooled. The pyridine hydrochloride that precipitated was filtered off, the filtrate was washed with water (3 × 50 ml) and 5% aqueous sodium bicarbonate (3 × 50 ml), and dried with $CaCl_2$. The drier was filtered off, and the solvent was removed. Compounds **IIIa**, **IIIf**, and **IIIm**–**IIIw** were purified by low-temperature crystallization from 96% ethanol and compounds **IIIb**–**IIIe** and **IIIg**–**IIIl**, by column chromatography on alumina (Brockmann activity grade II, neutral, 100–160 μm), eluent hexane.

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