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N-Heterocyclic Carbenes: IX.* Oxidative Esterification of Aromatic Aldehydes with Arylboronic Acids Catalyzed by *N*-Heterocyclic Carbenes

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Abstract—Aromatic aldehydes reacted with arylboronic acids under catalysis by *N*-heterocyclic carbene and oxic conditions to produce phenol esters.

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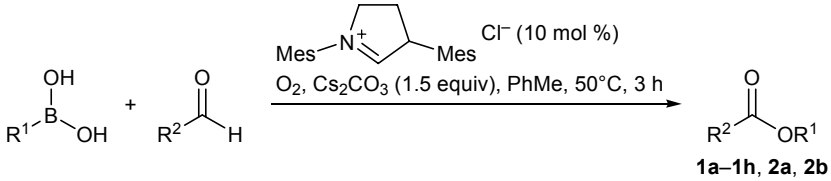
Esterification with the aid of *N*-heterocyclic carbenes (NHCs) is now a widely recognized synthetic method of organocatalysis [2–4]. We have found no data on the use of NHCs in esterification reactions in the Russian scientific literature, but examples of NHC-catalyzed transesterification have been reported [5]. The goal of the present work was to determine the scope of application of oxidative esterification.

Esterification of aldehydes with phenols catalyzed by NHCs via intramolecular redox reaction has recently been reported. These reactions are typical of aldehydes containing oxidizing groups, such as α-halo aldehydes [6, 7], 2,3-epoxy aldehydes [8], and α,β-unsaturated aldehydes [9]. Later it was shown that not only electron-withdrawing group in the substrate but also atmospheric oxygen can act as oxidant in NHC-catalyzed esterification, which made it possible to extend the series of aldehydes capable of reacting in this way [10, 11]. However, such aerobic esterification turned out to be inapplicable to phenols. An alternative route is the synthesis of phenol esters from arylboronic acids [12]. For this purpose, palladium complexes with NHCs were used as catalyst. The procedure ensured preparation of phenol esters from aromatic aldehydes and arylboronic acids under oxic conditions, but the yields were low. Gois et al. [13, 14] succeeded in raising the yield to 92% by replacing palladium(II) by iron(III). As shown subsequently in [15, 16], iron is not necessary to catalyze the reaction since *N*-heterocyclic carbenes themselves are active as organocatalysts.

We have synthesized 1-naphthyl and 4-tolyl esters in 25–63% yields by oxidative esterification, i.e., by NHC-catalyzed reaction of 4-methylphenyl- or naphthalen-1-ylboronic acid with aromatic aldehydes (Scheme 1). Taking into account published data [15, 16], as NHC catalyst we selected 1,3-dimesityl-4,5-dihydroimidazol-2-ylidene which was generated *in situ* from 1,3-dimesityl-4,5-dihydroimidazolium chloride [17] by the action of cesium carbonate. The reaction mechanism was discussed previously [15, 16]; Scheme 2 shows a simplified version.

Scheme 1.

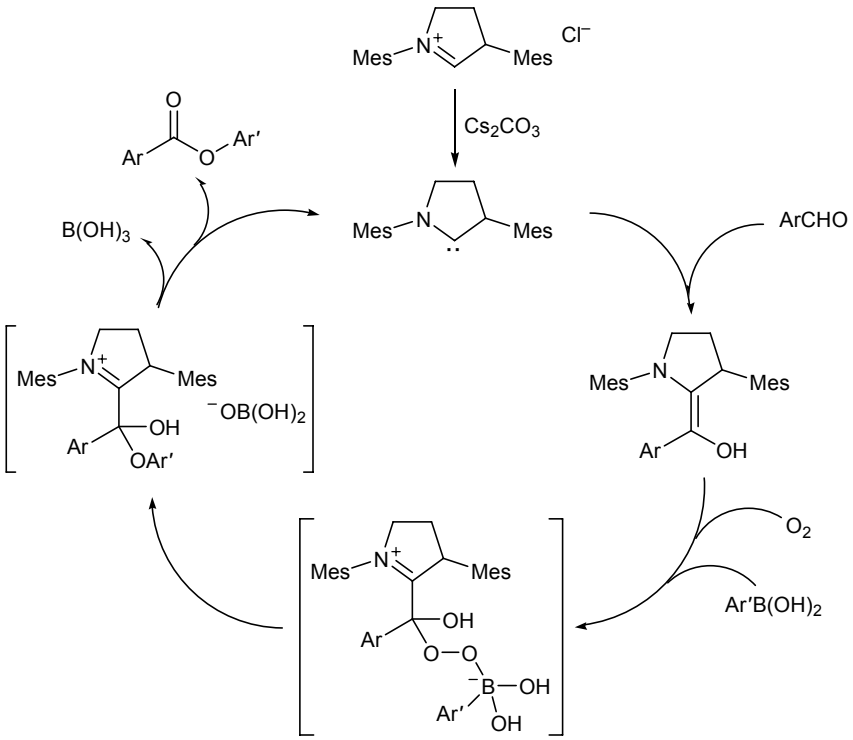


1a–1h, **2a**, **2b**

1, R^1 = 4-MeC_6H_4, R^2 = pyridin-2-yl (**a**), pyridin-3-yl (**b**), pyridin-4-yl (**c**), 2-thienyl (**d**), PhCH=CH (**e**), 3,4-$(MeO)_2C_6H_3$ (**f**), 3,4,5-$(MeO)_3C_6H_2$ (**d**), ferrocenyl (**h**); **2**, R^1 = 1-naphthyl, R^2 = pyridin-3-yl (**a**), ferrocenyl (**b**).

* For communication VIII, see [1].

Scheme 2.



Different pyridinecarbaldehydes were reacted with 4-methylphenylboronic acid. The position of the heteroatom with respect to the aldehyde group did not affect the reactivity, and the conversion of isomeric pyridine-2-, -3-, and -4-carbaldehydes was about ~90% (according to the GC/MS data). Pyridinecarbaldehydes may be regarded as electron-deficient. There are published data, according to which electron-deficient aldehydes are less reactive in oxidative esterification [15, 16, 18], while the opposite was stated in [14]. We have not revealed a statistically significant difference in the reactivities of electron-deficient and electron-rich aldehydes. The yields of the corresponding esters from thiophene-2-carbaldehyde, 3,4-dimethoxybenzaldehyde, and 3,4,5-trimethoxybenzaldehyde (98, 100, and 85%, respectively) were comparable with those obtained in the reactions with pyridinecarbaldehydes (92, 90, 89%). 3,4-Dimethoxybenzaldehyde turned out to be the most reactive. In the reaction with thiophene-2-carbaldehyde we isolated 22% of 1,2-dithienylethane-1,2-dione as by product. Cinnamaldehyde and ferrocenecarbaldehyde also reacted with arylboronic acids in the presence of NHC; in the latter case, the temperature should be raised to 70°C. Among the examined aldehydes, only vanillin and salicylaldehyde failed to react. Taking into account high reactivity of 3,4-dimethoxybenzaldehyde, we presumed that the presence of a phenolic hydroxy group makes vanillin and salicylaldehyde non-reactive.

As stated in [13, 14, 16], steric hindrances are essential for reactions with arylboronic acids. To verify this statement we examined the reactions of pyridine-3-carbaldehyde and ferrocenecarbaldehyde with naphthalen-1-ylboronic acid and revealed no reduction of the reactivity. Naphthalen-1-yl ferrocenecarboxylate was synthesized by us for the first time.

In summary, we have developed a simple preparative procedure for the synthesis of aryl arenecarboxylates and estimated its application scope.

EXPERIMENTAL

The IR spectra were recorded on a Bruker IFS 66ps spectrometer with Fourier transform from samples dispersed in mineral oil. The ^{1}H and ^{13}C NMR spectra were recorded from solutions in $CDCl_3$ on a Varian Mercury Plus 300 instrument at 300 and 75 MHz, respectively, using hexamethyldisiloxane (^{1}H) or solvent signals as reference. The melting points were determined on a PTP melting point apparatus. The products were isolated by column chromatography on Silicagel 60 (Alfa Aesar, 0.032–0.070 mm) using petroleum ether–ethyl acetate [gradient elution, 20:1 to 1:1 (**1b**, **1c**), to 3:1 (**1a**, **2a**), to 5:1 (**1f**), to 10:1 (**1g**, **1h**, **2b**);

20:1 (**1d**, **1e**)]. The purity of the isolated compounds was checked by GC/MS on an Agilent Technologies 6890N/5975B GC/MS system (HP-5MS capillary column, 30000×0.25 mm; injector temperature 260°C, oven temperature programming at a rate of 20 to 40 deg/min; carrier gas helium, flow rate 1 mL/min; electron impact, 70 eV).

4-Methylphenylboronic acid and naphthalen-1-ylboronic acid were synthesized as described in [19].

General procedure for the synthesis of esters 1 and 2. A mixture of 17.1 mg (0.05 mmol) of 1,3-bis-(2,4,6-trimethylphenyl)-4,5-dihydro-3*H*-imidazol-1-ium chloride, 244 mg (0.75 mmol) of Cs_2CO_3, 0.65 mmol of the corresponding aldehyde, 0.5 mmol of arylboronic acid, and 5 mL of toluene was stirred for 3 h at 50°C (at 70°C in the synthesis of **1h**). When the reaction was complete, a sample of the reaction mixture was withdrawn, dried over $MgSO_4$, diluted with methylene chloride to a product concentration of ~1 mg/mL, and analyzed by GC/MS. The solvent was distilled off from the reaction mixture on a rotary evaporator, and the residue was purified by column chromatography.

4-Methylphenyl pyridine-2-carboxylate (1a) was synthesized from 0.062 mL of pyridine-2-carbaldehyde and 68 mg of 4-methylphenylboronic acid. Yield 61 mg (58%; 92% according to GC/MS), gray crystals, mp 76–79°C [20], R_f 0.56 (petroleum ether–ethyl acetate, 1:1). IR spectrum, ν, cm^{-1}: 1750, 1665, 1580, 1508, 1303, 1278, 1236, 1189, 1167, 1113, 1072, 1047, 1017, 993, 879, 817, 805, 750, 701, 687, 618, 514. ^{1}H NMR spectrum, δ, ppm: 2.36 s (3H, Me), 7.12 d (2H, 2′-H, 6′-H, ^3J = 8.4 Hz), 7.22 d (2H, 3′-H, 5′-H, ^3J = 8.4 Hz), 7.55 d.d.d (1H, 5-H, ^3J = 10.5, 7.8, ^4J = 1.2 Hz), 7.91 d.d.d (1H, 4-H, ^3J = 7.8, 7.8, ^4J = 1.5 Hz), 8.27 d (1H, 3-H, ^3J = 7.8 Hz), 8.84 d.d (1H, 6-H, ^3J = 7.8, ^4J = 1.5 Hz). Mass spectrum, *m*/*z* (I_{rel}, %): 213 (1) $[M]^{+\cdot}$, 170 (7) $[PyTol + 1]^+$, 169 (58) $[PyTol]^+$, 107 (7) $[TolO]^+$, 106 (31) $[PyCO]^+$, 79 (10) $[PyH]^+$, 78 (100) $[Py]^+$, 77 (14) $[Py – H]^+$, 52 (7), 51 (18).

4-Methylphenyl pyridine-3-carboxylate (1b) was synthesized from 0.060 mL of pyridine-3-carbaldehyde and 68 mg of 4-methylphenylboronic acid. Yield 66 mg (62%; 90%, GC/MS), white crystals, mp 64–65°C (published data [21]: mp 62–63°C), R_f 0.38 (chloroform–ethyl acetate, 1:1). IR spectrum, ν, cm^{-1}: 1729, 1688, 1589, 1507, 1422, 1323, 1279, 1248, 1194, 1161, 1127, 1093, 1077, 1037, 1018, 971, 941, 876, 758, 702, 687, 524. ^{1}H NMR spectrum, δ, ppm: 2.37 s (3H, Me), 7.09 d (2H, 2′-H, 6′-H, ^3J = 8.4 Hz), 7.23 d (2H, 3′-H, 5′-H, ^3J = 8.4 Hz), 7.53 d.d (1H, 5-H, ^3J = 7.5, 4.8 Hz), 8.44 d (1H, 4-H, ^3J = 7.5 Hz), 8.84 d (1H, 6-H, J = 4.8 Hz), 9.39 s (2-H). Mass spectrum, *m*/*z* (I_{rel}, %): 213 (15) $[M]^{+\cdot}$, 107 (10) $[TolO]^+$, 106 (100) $[PyCO]^+$, 78 (46) $[Py]^+$, 77 (10) $[Py – H]^+$, 51 (15).

4-Methylphenyl pyridine-4-carboxylate (1c) was synthesized from 0.062 mL of pyridine-4-carbaldehyde and 68 mg of 4-methylphenylboronic acid. Yield 60 mg (56%; 89%, GC/MS), white crystals, mp 69–71°C [22], R_f 0.55 (petroleum ether–ethyl acetate, 1:1). IR spectrum, ν, cm^{-1}: 1743, 1410, 1325, 1272, 1218, 1196, 1171, 1098, 1085, 1063, 1025, 880, 848, 830, 751, 670, 510. ^{1}H NMR spectrum, δ, ppm: 2.37 s (3H, Me), 7.09 d (2H, 2′-H, 6′-H, ^3J = 8.4 Hz), 7.23 d (2H, 3′-H, 5′-H, ^3J = 8.4 Hz), 8.01 d.d (2H, 3-H, 5-H, ^3J = 4.8, ^4J = 1.5 Hz), 8.85 d (2H, 2-H, 6-H, ^3J = 4.8 Hz). Mass spectrum, *m*/*z* (I_{rel}, %): 213 (18) $[M]^{+\cdot}$, 107 (13) $[TolO]^+$, 106 (100) $[PyCO]^+$, 79 (6) $[PyH]^+$, 78 (52) $[Py]^+$, 77 (13) $[Py – H]^+$, 51 (24), 50 (7).

4-Methylphenyl thiophene-2-carboxylate (1d) was synthesized from 0.061 mg of thiophene-2-carbaldehyde and 68 mg of 4-methylphenylboronic acid. Yield 126 mg (62%; 98%, GC/MS), yellow powder, mp 77–83°C (published data [23]: mp 85–87°C), R_f 0.55 (petroleum ether–ethyl acetate, 7:3). IR spectrum, ν, cm^{-1}: 3108, 3099, 3090, 3080, 1713, 1648, 1609, 1595, 1525, 1313, 1255, 1224, 1210, 1197, 1165, 1102, 1083, 1063, 1022, 954, 866, 854, 841, 820, 792, 744, 698, 663, 636, 567, 517, 500. ^{1}H NMR spectrum, δ, ppm: 2.34 s (3H, Me), 7.07 d (2H, H_{arom}), 7.13 d.d (1H, 4-H, ^3J = 5.1, 3.6 Hz), 7.18 d (2H, H_{arom}), 7.61 d.d (1H, 3-H, ^3J = 5.1, ^4J = 1.2 Hz), 7.94 d.d (1H, 5-H, ^3J = 3.6, ^4J = 1.2 Hz). ^{13}C NMR spectrum, δ_C, ppm: 20.84, 121.25, 127.92, 129.91, 133.01, 133.28, 134.48, 135.55, 148.32, 160.70. Mass spectrum, *m*/*z* (I_{rel}, %): 218 (10) $[M]^{+\cdot}$, 112 (6) $[(C_4H_3S)CO + 1]^+$, 111 (100) $[(C_4H_3S)CO]^+$, 83 (6) $[C_4H_3S]^+$, 77 (6).

4-Methylphenyl (2*E*)-3-phenylprop-2-enoate (1e) was synthesized from 0.082 mL of cinnamaldehyde and 68 mg of 4-methylphenylboronic acid. Yield 29 mg (25%; 81%, GC/MS), gray powder, mp 84–86°C (published data [24]: mp 84–85°C), R_f 0.50 (petroleum ether–ethyl acetate, 7:3). IR spectrum, ν, cm^{-1}: 3063, 1731, 1651, 1635, 1615, 1593, 1576, 1557, 1506, 1496, 1332, 1308, 1278, 1198, 1179, 1166, 1138, 1102, 1027, 1016, 991, 974, 945, 860, 846, 812, 759, 705, 678, 578, 527, 507, 486, 435. ^{1}H NMR spectrum, δ, ppm: 2.34 s (3H, Me), 6.61 d (β-CH=, ^3J = 16.2 Hz), 7.04 d (2H, H_{arom}, ^3J = 8.4 Hz), 7.19 (2H, H_{arom}, ^3J =

8.4 Hz), 7.39–7.41 m (3H, H_{arom}), 7.55–7.58 m (2H, H_{arom}), 7.85 d (α-CH=, ^3J = 16.2 Hz). Mass spectrum, *m*/*z* (I_{rel}, %): 238 (3) $[M]^{+\cdot}$, 132 (10) $[PhCHCHCO + 1]^+$, 131 (100) $[PhCHCHCO]^+$, 103 (30) $[PhCHCH]^+$, 77 (24) $[Ph]^+$.

4-Methylphenyl 3,4-dimethoxybenzoate (1f) was synthesized from 108 mg of 3,4-dimethoxybenzaldehyde and 68 mg of 4-methylphenylboronic acid. Yield 85 mg (62%; 100%, GC/MS), gray crystals, mp 125–131°C [25], R_f 0.32 (petroleum ether–ethyl acetate, 7:3). IR spectrum, ν, cm^{-1}: 3017, 1725, 1600, 1509, 1416, 1350, 1290, 1272, 1248, 1217, 1195, 1189, 1168, 1143, 1102, 1086, 1023, 962, 945, 916, 879, 863, 825, 787, 769, 757, 738, 632, 517, 502. ^1H NMR spectrum, δ, ppm: 2.35 s (3H, Me), 3.93 s (3H, OMe), 3.94 s (3H, OMe), 6.91 d (1H, 5-H, ^3J = 8.4 Hz), 7.06 d (2H, H_{arom}, ^3J = 8.4 Hz), 7.19 d (2H, H_{arom}, ^3J = 8.4 Hz), 7.65 d (1H, 2-H, ^4J = 2.1 Hz), 7.83 d.d (1H, 6-H, ^3J = 8.4, ^4J = 2.1 Hz). Mass spectrum, *m*/*z* (I_{rel}, %): 272 (6) $[M]^{+\cdot}$, 166 (10) $[(MeO)_2C_6H_3CO + 1]^+$, 165 (100) $[(MeO)_2C_6H_3CO]^+$, 137 (6) $[(MeO)_2C_6H_3]^+$, 79 (7), 77 (8).

4-Methylphenyl 3,4,5-trimethoxybenzoate (1g) was synthesized from 128 mg of 3,4,5-trimethoxybenzaldehyde and 68 mg of 4-methylphenylboronic acid. Yield 68 mg (45%; 77%, GC/MS), gray crystals, mp 85–88°C (published data [26]: mp 88–89°C), R_f 0.43 (petroleum ether–ethyl acetate, 7:3). IR spectrum, ν, cm^{-1}: 1727, 1587, 1507, 1415, 1337, 1230, 1215, 1189, 1169, 1159, 1129, 1031, 1020, 1003, 943, 928, 915, 866, 844, 824, 786, 773, 758, 696, 503. ^1H NMR spectrum, δ, ppm: 2.36 s (3H, Me), 3.93 s (9H, OMe), 7.07 d (2H, H_{arom}, ^3J = 8.4 Hz), 7.16 d (2H, H_{arom}, ^3J = 8.4 Hz), 7.44 s (2H, H_{arom}). Mass spectrum, *m*/*z* (I_{rel}, %): 302 (6) $[M]^{+\cdot}$, 196 (11) $[(MeO)_3C_6H_2CO + 1]^+$, 195 (100) $[(MeO)_3C_6H_2CO]^+$, 77 (7).

4-Methylphenyl ferrocenecarboxylate (1h) was synthesized from 139 mg of ferrocenecarbaldehyde and 68 mg of 4-methylphenylboronic acid. Yield 135 mg (45%; 85%, GC/MS), red crystals, mp 93–94°C (published data [27]: mp 90–94°C), R_f 0.43 (petroleum ether–ethyl acetate, 7:3). IR spectrum, ν, cm^{-1}: 1732, 1273, 1215, 1192, 1163, 1097, 1022, 1016, 1000, 911, 860, 829, 815, 783, 764, 505, 485. ^1H NMR spectrum, δ, ppm: 2.35 s (3H, Me), 4.28 s (5H, Fc), 4.46 m (2H, Fc), 4.94 m (2H, Fc), 7.04 d (2H, H_{arom}, ^3J = 8.4 Hz), 7.19 d (2H, H_{arom}, ^3J = 8.4 Hz). Mass spectrum, *m*/*z* (I_{rel}, %): 321 (15) $[M + 1]^{+\cdot}$, 320 (69) $[M]^{+\cdot}$, 228 (21) $[FcCO_2]^+$, 214 (14) $[FcCO + 1]^+$, 213 (100) $[FcCO]^+$, 211 (6) $[FcCO - 2]^+$, 200 (6), 186 (7) $[Fc + 1]^+$, 185 (48) $[Fc]^+$, 129 (30), 128 (11) $[CpCp]^+$, 121 (23) $[FeCp]^+$, 77 (7), 56 (11) $[Fe]^+$.

Naphthalen-1-yl pyridine-3-carboxylate (2a) [28] was synthesized from 0.061 mL of pyridine-3-carbaldehyde and 86 mg of naphthalen-1-ylboronic acid. Yield 72 mg (58%; 100%, GC/MS), brown viscous liquid, R_f 0.17 (petroleum ether–ethyl acetate, 7:3). IR spectrum, ν, cm^{-1}: 3057, 1742, 1674, 1590, 1576, 1508, 1420, 1390, 1327, 1273, 1256, 1224, 1194, 1155, 1098, 1064, 1039, 1018, 871, 793, 770, 700, 590, 559, 508, 418. ^1H NMR spectrum, δ, ppm: 7.38 d (1H, 2′-H, ^3J = 7.5 Hz), 7.39–7.53 m (4H, 3′-H, 5-H, 6′-H, 7′-H), 7.79 d (1H, 4′-H, ^3J = 8.4 Hz), 7.88–7.91 m (2H, 4-H, 5′-H), 8.56 d.d (1H, 8′-H, ^3J = 8.1, ^4J = 1.8 Hz), 8.91 m (1H, 6-H), 9.54 s (1H, 2-H). Mass spectrum, *m*/*z* (I_{rel}, %): 249 (21) $[M]^{+\cdot}$, 115 (21) $[indene]^{+\cdot}$, 107 (6) $[PyCO + 1]^+$, 106 (100) $[PyCO]^+$, 78 (39) $[Py]^+$, 51 (11).

Naphthalen-1-yl ferrocenecarboxylate (2b) was synthesized from 139 mg of ferrocenecarbaldehyde and 86 mg of naphthalen-1-ylboronic acid. Yield 112 mg (63%; 77%, GC/MS), red crystals, mp 92–94°C, R_f 0.58 (petroleum ether–ethyl acetate, 7:3). IR spectrum, ν, cm^{-1}: 3099, 3077, 1732, 1680, 1633, 1599, 1575, 1413, 1351, 1274, 1258, 1224, 1154, 1105, 1054, 1038, 1027, 1011, 970, 961, 915, 897, 866, 824, 783, 767, 759, 668, 627, 606, 596, 561, 541, 527, 505, 482, 459, 451, 422. ^1H NMR spectrum, δ, ppm: 4.33 s (5H, Fc), 4.51 m (2H, Fc), 5.06 m (2H, Fc), 7.35 d (1H, 2-H, ^3J = 7.8 Hz), 7.48–7.53 m (3H, 3-H, 6-H, 7-H), 7.73 d (1H, 4-H, ^3J = 8.1 Hz), 7.86 m (1H, 5-H), 8.05 m (1H, 8-H). ^{13}C NMR spectrum, δ_C, ppm: 69.99, 70.64, 70.72, 71.97, 118.16, 121.23, 125.44, 125.62, 126.25, 126.37, 127.13, 128.01, 134.67, 146.63, 170.34. Mass spectrum, *m*/*z* (I_{rel}, %): 357 (8) $[M + 1]^{+\cdot}$, 356 (37) $[M]^{+\cdot}$, 264 (10) $[M - TolH]^+$, 214 (14) $[FcCO + 1]^+$, 213 (100) $[FcCO]^+$, 211 (6) $[FcCO - 2]^+$, 207 (7), 198 (15) $[FcCH]^+$, 185 (41) $[Fc]^+$, 129 (28) $[CpCpH]^+$, 128 (8) $[CpCp]^+$, 121 (17) $[FeCp]^+$, 115 (17) $[indene]^{+\cdot}$, 56 (7) $[Fe]^+$. Found, %: C 70.87; H 4.52. $C_{21}H_{16}FeO_2$. Calculated, %: C 70.81; H 4.53. *M* 356.20.

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