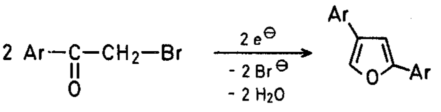
Electrochemical Synthesis of 2,4-Diarylfurans

Fructuoso BARBA, M. Desamparados VELASCO, Antonio GUIRADO

Department of Organic Chemistry, Faculty of Science, University of Murcia, Murcia, Spain

The electrochemical reduction of carbon-halogen bonds has been studied in detail[1]. Unfortunately, however, a significant portion of the published work has dealt with the mechanism of the reduction rather than with its utility in organic synthesis.

In the present paper, we report on the electrochemical reduction of phenacyl bromides (**1**) to give 2,4-diarylfurans (**2**). Our results are summarised below.



1 **2**

The present method is more convenient in terms of yield, simplicity, and availability of starting materials than previously reported methods[2,3].

2,4-Diarylfurans 2; General Procedure:

Anode: platinum.

Anolyte: lithium perchlorate (4 mmol) in dry dimethylformamide (20 ml).

Cathode: mercury pool.

Catholyte: phenacyl bromide **1** (10 mmol) and lithium perchlorate (6 mmol) in dry dimethylformamide (30 ml).

Electrolysis cell: divided cell thermostated at 20 °C, equipped with a magnetic stirrer containing a piece of glass tubing with glass frit of medium porosity at one end to serve as auxiliary electrode (cathode) compartment.

The electrolysis is carried out under a controlled potential of − 1 V (vs saturated calomel electrode) using an Amel potentiostat model 557 with an electronic integrator Amel model 558.

Solid sodium thiosulphate (2 g) and sodium carbonate (3 g) are added to the anode compartment for *in situ* neutralisation of the bromine and acid generated. The electrolysis is continued until the current decreases to less then 1% of the starting value. The amount of charge passed is the equivalent of 1 electron consumed per molecule of substrate. The cathode solution is worked up by evaporation of the solvent to dryness at 60 °C under reduced pressure. The residue is extracted with water (2 x 50 ml) to remove inorganic salts. The residue is washed with cold methanol and recrystallised from a suitable solvent to give the product **2**.

2,4-Diphenylfuran (**2a**; Ar = C_6H_5); yield: 0.881 g (80%); colourless flakes from methanol; m.p. 109-110 °C (Ref.[4], m.p. 109-111 °C); intense blue fluorescence in methanol and concentrated sulphuric acid.

$C_{16}H_{12}O$	calc.	C 87.25	H 5.49
(220.3)	found	87.40	5.48

M.S.: m/e (relative intensity) = 220 (M^+, 100), 191 (94), 165 (25), 115 (14).

I.R. (KBr): ν = 1448, 1195, 1143, 925, 911, 810, 740, 690 cm^{-1}.

2,4-Bis[4-methoxyphenyl]furan (**2b**; Ar = 4-H_3CO—C_6H_4); yield: 1.054 g (75%); colourless flakes from methanol; m.p. 191-192 °C; intense blue fluorescence in methanol and green in concentrated sulphuric acid.

$C_{18}H_{16}O_3$	calc.	C 77.12	H 5.75
(280.3)	found	77.27	5.74

M.S.: m/e (relative intensity) = 280 (M^+, 100), 265 (13), 251 (17), 236 (44), 194 (19), 178 (18), 165 (52).

I.R. (KBr): ν = 1510, 1450, 1306, 1250, 1182, 1030, 920, 813 cm^{-1}.

2,4-Bis[4-biphenylyl]furan (**2c**; Ar = 4-C_6H_5—C_6H_4); yield: 1.242 g (67%); colourless flakes from chloroform; m.p. 267-268 °C; intense

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[1] G. Szeimies, R. Huisgen, *Chem. Ber.* **99**, 491 (1966).

[2] V. K. Antonov, *Zh. Obshch. Khim.* **29**, 1132 (1959); *C. A.* **54**, 1480 (1960).

[3] K.-D. Gundermann, G. Holtmann, H.-J. Rose, H. Schulze, *Chem. Ber.* **93**, 1632 (1960).

[4] K.-D. Gundermann, K. Burzin, F.-J. Sprenger, H. Schulze, *Chem. Ber.* **105**, 312 (1972).

[5] H. Brintzinger, A. Scholz, *Chem. Ber.* **83**, 141 (1950).

[6] T. Wagner-Jauregg, *Helv. Chim. Acta* **44**, 1237 (1961).

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blue fluorescence in methanol and yellow in concentrated sulphuric acid.

$C_{28}H_{20}O$	calc.	C 90.29	H 5.41	
(372.5)	found	90.49	5.40	

M.S.: *m/e* (relative intensity) = 372 (M^+, 100), 343 (32), 267 (11), 265 (14), 189 (9), 166 (9), 153 (9).

I.R. (KBr): ν = 1489, 1418, 917, 842, 809, 768, 760, 723, 690 cm^{-1}.

2,4-Bis[4-chlorophenyl]furan (**2d**; Ar = 4-Cl—C_6H_4); yield: 1.052 g (73%); colourless needles from methanol; m.p. 127–128 °C (Ref.[5], m.p. 128–129 °C); intense blue fluorescence in methanol and concentrated sulphuric acid.

$C_{16}H_{10}Cl_2O$	calc.	C 66.45	H 3.49	
(289.2)	found	66.60	3.50	

M.S.: *m/e* (relative intensity) = 290 ($M^+ + 2$, 59), 288 (M^+, 100), 261 (21), 259 (38), 227 (27), 225 (82), 190 (64), 94 (36).

I.R. (KBr): ν = 1485, 1412, 1194, 1000, 920, 903, 835, 829, 811, 800, 769 cm^{-1}.

2,4-Bis[4-bromophenyl]furan (**2e**; Ar = 4-Br—C_6H_4); yield: 1.209 g (64%); colourless flakes from methanol; m.p. 159–160 °C (Ref.[6], m.p. 160 °C); intense violet fluorescence in methanol and blue in concentrated sulphuric acid.

$C_{16}H_{10}Br_2O$	calc.	C 50.83	H 2.67	
(378.1)	found	50.73	2.67	

M.S.: *m/e* (relative intensity) = 380 ($M^+ + 2$, 25), 378 (M^+, 49), 376 (26), 351 (5), 349 (10), 347 (5), 218 (30), 189 (100), 163 (18), 95 (44).

I.R. (KBr): ν = 1484, 1413, 1074, 1008, 912, 830, 800, 766 cm^{-1}.

2,4-Bis[4-nitrophenyl]furan (**4f**; Ar = 4-O_2N—C_6H_4); yield: 1.008 g (65%); yellow crystals from chloroform; m.p. 225–226 °C; no fluorescence.

$C_{16}H_{10}N_2O_5$	calc.	C 61.94	H 3.25	N 9.03
(310.3)	found	62.06	3.24	9.01

M.S.: *m/e* (relative intensity) = 310 (M^+, 73), 280 (25), 279 (20), 234 (18), 218 (21), 206 (15), 189 (100), 178 (32), 163 (22), 152 (20), 95 (33).

I.R. (KBr): ν = 1618, 1604, 1512, 1347, 1113, 987, 971, 861, 758 cm^{-1}.

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[1] *Techniques of Electroorganic Synthesis*, Part II, N. L. Weinberg, Ed., John Wiley & Sons, Inc., New York, 1975, p. 170–192.

[2] E. Yoshisato, S. Tsutsumi, *J. Chem. Soc. Chem. Commun.* **1968**, 33.

[3] H. J. Seebald, W. Schunack, *J. Chromatogr.* **74**, 129 (1972).

[4] *Beilsteins Handbuch der Organischen Chemie*, Band XVII, Springer-Verlag, Berlin, 1933, 1952.

[5] W. Ried, W. Bodenstedt, *Justus Liebigs Ann. Chem.* **679**, 77 (1964).

[6] T. Shirafuji, Y. Yamamoto, H. Nozaki, *Tetrahedron Lett.* **1969**, 4097.

0039-7881/81/0832-0626 $ 03.00