

## Polyfluoroarenes. Part XVIII.<sup>1</sup> Some Reactions of 2,3,4,5,6-Pentafluorobiphenyl and Related Compounds

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2,3,4,5,6-Pentafluorobiphenyl reacts with boiling fuming nitric acid to give 2,4-dinitrobenzoic acid, but under mild conditions the 2'-, 3'-, and 4'-nitro-derivatives are formed in the ratio 3·4 : 1·0 : 8·7. All three nitro-compounds may be synthesised unambiguously by mixed Ullmann reactions. 2,3,4,5,6-Pentafluoro-3'-nitrobiphenyl is also isolated following the oxidation of pentafluorophenylhydrazine in nitrobenzene, and reacts with hot fuming nitric acid to give 3,4-dinitrobenzoic acid. Syntheses of 2,3,5,6-tetrafluoro-4-hydroxy-, -4-methoxy-, and -4-iodobiphenyl are also described; irradiation of the iodo-compound in benzene gives 2',3',5',6'-tetrafluoro-*p*-terphenyl, and its Ullmann condensation provides a good route to 2',2'',3',3'',5',5'',6',6''-octafluoro-*p*-quaterphenyl.

2,3,4,5,6-PENTAFLUOROBIPHENYL may be obtained in good yield either by the reaction of pentafluorophenyl radicals with benzene<sup>2,3</sup> or by nucleophilic displacement of fluoride from hexafluorobenzene by the action of phenyl-lithium.<sup>4,5</sup> It is also formed by the attack of phenyl radicals on hexafluorobenzene.<sup>6</sup> The biphenyl

<sup>1</sup> Part XVII, J. M. Birchall, R. N. Haszeldine, and M. E. Jones, *J. Chem. Soc. (C)*, 1971, 1343.

<sup>2</sup> J. M. Birchall, R. N. Haszeldine, and A. R. Parkinson, *J. Chem. Soc.*, 1962, 4966.

<sup>3</sup> J. M. Birchall, R. N. Haszeldine, R. Hazard, and A. W. Wakalski, *J. Chem. Soc. (C)*, 1967, 47.

undergoes nucleophilic displacement of fluoride at the 4-position in its reactions with hydrazine hydrate, ammonia, and sodium hydrosulphide,<sup>5</sup> and electrophilic bromination, nitration, and methylation in the hydrocarbon ring have also been studied.<sup>7</sup> Studies, carried

<sup>4</sup> L. A. Wall, W. J. Pummer, J. E. Fearn, and J. M. Antonucci, *J. Res. Nat. Bur. Stand.*, 1963, 67A, 481.

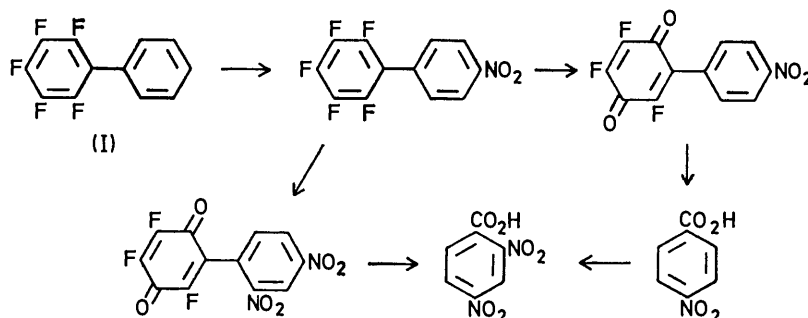
<sup>5</sup> M. T. Chaudhry and R. Stephens, *J. Chem. Soc.*, 1963, 4281.

<sup>6</sup> P. A. Claret, G. H. Williams, and J. Coulson, *J. Chem. Soc. (C)*, 1968, 341.

<sup>7</sup> P. J. N. Brown, M. T. Chaudhry, and R. Stephens, *J. Chem. Soc. (C)*, 1969, 2747.

out some years ago, of the oxidative stability of the biphenyl, of some additional nucleophilic and electrophilic displacement reactions, and of the properties of some of its derivatives are described here.

**Oxidation Reactions.**—2,3,4,5,6-Pentafluorobiphenyl does not react with refluxing aqueous potassium permanganate during 3 h nor with potassium permanganate in boiling aqueous acetone during 4 h. Like biphenyl itself,<sup>8</sup> the pentafluoro-compound (I) undergoes nitration in fuming nitric acid at room temperature (see later), but under reflux conditions this reagent causes complete breakdown of the fluoroaromatic ring during 24 h and yields 2,4-dinitrobenzoic acid (48%). Hexafluorobenzene itself undergoes oxidation to fluoranil in nitric acid,<sup>9</sup> but since mild conditions lead to mononitration of the biphenyl, it seems likely that oxidation of the latter follows mainly the paths outlined (Scheme 1).



SCHEME 1

Chromium trioxide in refluxing acetic acid, which is effective in oxidising 2,3,4,5,6-pentafluorotoluene to pentafluorobenzoic acid in low yield,<sup>10</sup> also breaks down the fluoroaromatic ring in the biphenyl (I), to give benzoic acid [8% based on biphenyl transformed (48% during 10 h)].

**Pentafluoronitrobiphenyls.**—Brown, Chaudhry, and Stephens have reported that treatment of 2,3,4,5,6-pentafluorobiphenyl with fuming nitric and concentrated sulphuric acid in refluxing glacial acetic acid gives only pentafluoro-2'-nitro- and pentafluoro-4'-nitro-biphenyl in the approximate ratio of 1:4.<sup>7</sup> No nitration occurs when the pentafluoro-compound is heated with only fuming nitric acid in glacial acetic acid at 90° for 1 h (conditions which easily nitrate biphenyl itself<sup>8</sup>), but in the absence of acetic acid, fuming nitric acid reacts spontaneously at room temperature and gives all three pentafluoronitrobiphenyls [(II), (III), and (IV)], isolated by crystallisation techniques in 71% total yield in the ratio 2':3':4' ≡ 3.4:1.0:8.7.

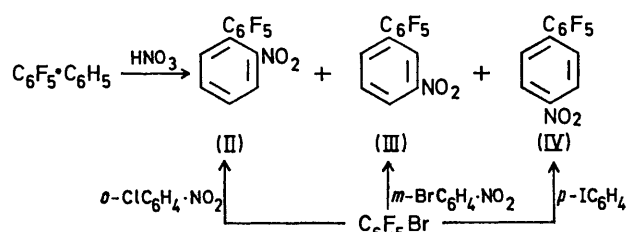
All three compounds were identified by unambiguous Ullmann synthesis as shown (Scheme 2). The pre-

viously reported failure of *p*-bromonitrobenzene to undergo a successful Ullmann condensation with bromopentafluorobenzene<sup>7</sup> is confirmed.

The comparable, but easier, nitration of biphenyl itself yields only 2- and 4-nitrobiphenyl,<sup>8</sup> and the formation of a significant proportion of the 3'-nitro-compound (III) during the nitration described here may be attributed to the  $-I$  effect of the fluorine atoms in the pentafluorophenyl ring,<sup>11a</sup> which must exert a destabilising influence on the intermediates involved in nitration at the 2'- and 4'-positions. The strongly *meta*-directing and deactivating influence of a trifluoromethyl substituent on electrophilic aromatic substitution is well established.<sup>11b</sup>

Pentafluoro-3'-nitrobiphenyl is also the product most easily isolated (10% yield) following the oxidation with bleaching powder of pentafluorophenylhydrazine in

nitrobenzene. This reaction proceeds *via* formation of the pentafluorophenyl radical,<sup>2</sup> and the reaction with nitrobenzene of this radical generated from pentafluoroiodobenzene,<sup>3</sup> from decafluorobenzoyl peroxide,<sup>12</sup> or



SCHEME 2

from pentafluoroaniline and pentyl nitrite<sup>13</sup> has been described, but preparative separation of the isomeric products has not been attempted. Treatment of the 3'-nitrobiphenyl (III) with refluxing fuming nitric acid gives 3,4-dinitrobenzoic acid in high yield (73%); in view of the established directing effects of the aromatic substituents involved here, it seems likely that nitration

<sup>8</sup> H. Hübner and H. Lüddens, *Ber.*, 1875, **8**, 871; *Annalen*, 1881, **209**, 341.

<sup>9</sup> G. G. Yakobson, V. D. Shteingarts, and N. N. Vorozhtsov, *Zhur. Vsesoyuz. Khim. obshch. im. D. T. Mendeleeva*, 1964, **9**, 701.

<sup>10</sup> J. M. Birchall and R. N. Haszeldine, *J. Chem. Soc.*, 1961, 3719.

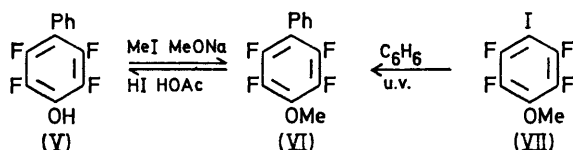
<sup>11</sup> (a) W. A. Sheppard, *J. Amer. Chem. Soc.*, 1970, **92**, 5419; (b) A. E. Pavlath and A. L. Leffler, 'Aromatic Fluorine Compounds,' Reinhold, New York, 1962, p. 32.

<sup>12</sup> P. H. Oldham and G. H. Williams, *J. Chem. Soc. (C)*, 1970, 1260.

<sup>13</sup> P. H. Oldham, G. H. Williams, and B. A. Wilson, *J. Chem. Soc. (C)*, 1971, 1094.

of the biphenyl in the 4'-position precedes fission of the pentafluorophenyl ring.

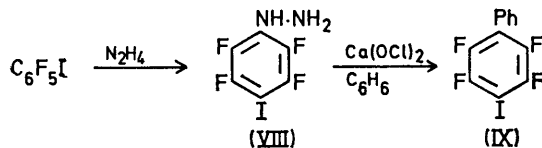
**Nucleophilic Substitution in Pentafluorobiphenyl.**—2,3,4,5,6-Pentafluorobiphenyl reacts with potassium hydroxide in refluxing *t*-butyl alcohol to give 2,3,5,6-tetrafluoro-4-hydroxybiphenyl (V) (39%)<sup>14</sup> and with sodium methoxide in refluxing methanol to give the corresponding methoxy-compound (VI) (80%). The fact that the nucleophile attacks the biphenyl at the same position in both these reactions is demonstrated by the interconversions shown (Scheme 3), although the



SCHEME 3

demethylation reaction has been achieved in only low yield. That the substituents both occupy the 4-position is established by unambiguous photochemical synthesis of the methoxybiphenyl (VI) (69% yield) from the known 2,3,5,6-tetrafluoro-4-iodoanisole (VII).

**2,3,5,6-Tetrafluoro-4-iodobiphenyl.**—The value of polyfluoroiodoarenes as intermediates for the synthesis of polyfluoropolyphenyls is now well established,<sup>3,15</sup> and the tetrafluoro-4-iodobiphenyl (IX) could provide a useful addition to the range of compounds available for the synthesis of these potentially useful polymeric systems. A small amount of the biphenyl is obtained as a by-product when tetrafluoro-1,4-di-iodobenzene is irradiated in an excess of benzene,<sup>15</sup> and it is possible that this reaction could be modified to provide tetrafluoro-4-iodobiphenyl in reasonable yield. However, a convenient synthesis is provided by the route shown in Scheme 4; an excellent yield (97%) of tetrafluoro-4-iodophenylhydrazine is obtained in the first step,<sup>16</sup> and the yield of biphenyl (IX) (38%) formed from the tetrafluoro-4-iodophenyl radical in the second step could probably be improved by the use of other oxidising agents.



SCHEME 4

Irradiation of the iodobiphenyl (IX) in benzene yields the expected 2',3',5',6'-tetrafluoro-*p*-terphenyl (60%), and the biphenyl undergoes a conventional Ullmann

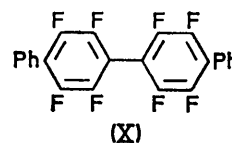
<sup>14</sup> Cf. J. M. Birchall and R. N. Haszeldine, *J. Chem. Soc.*, 1959, 13.

<sup>15</sup> J. M. Birchall, R. N. Haszeldine, and J. G. Speight, *J. Chem. Soc. (C)*, 1970, 2187.

<sup>16</sup> J. Burdon, P. L. Coe, C. R. Marsh, and J. C. Tatlow, *Tetrahedron*, 1966, **22**, 1183.

<sup>17</sup> N. A. Lange, 'Handbook of Chemistry,' McGraw-Hill, New York, 10th edn., 1961.

reaction at 200° in the presence of an excess of copper-bronze to give the octafluoroquaterphenyl (X) (77%).



## EXPERIMENTAL

**Oxidative Nitration of 2,3,4,5,6-Pentafluorobiphenyl.**—The biphenyl (5.0 g, 20.4 mmol)<sup>2,4,5</sup> and fuming nitric acid (40 ml; *d* 1.5) were heated under reflux for 24 h; the product was poured into water (50 ml), neutralised with sodium carbonate, and concentrated by distillation. Acidification of the solution, extraction with methylene chloride, and recrystallisation of the product from water gave 2,4-dinitrobenzoic acid (2.1 g, 48%), m.p. and mixed m.p. 179—181° (lit.,<sup>17</sup> 179—180°), identified by i.r. spectroscopy.

**Oxidation of 2,3,4,5,6-Pentafluorobiphenyl.**—Chromium trioxide (35.0 g, 350 mmol) in water (20 ml) and glacial acetic acid (12 ml) was added during 30 min to a stirred solution of the biphenyl (5.0 g, 20.4 mmol) in refluxing glacial acetic acid (65 ml). The mixture was then heated under reflux for 10 h, boiled with a solution of acetaldehyde (6 ml) in glacial acetic acid (15 ml) for 15 min, cooled, and poured into aqueous sodium chloride (50 ml). The products were extracted with methylene chloride and separated by extraction with aqueous ammonia to give 2,3,4,5,6-pentafluorobiphenyl (2.60 g, 52%) and benzoic acid (0.10 g, 8% based on biphenyl consumed), m.p. and mixed m.p. 120—121°. No oxidation products were detected following similar experiments in which the reaction times were 3 and 7.5 h.

**Ullmann Reactions.**—(a) **2,3,4,5,6-Pentafluoro-2'-nitrobiphenyl.** Bromopentafluorobenzene (10.0 g, 42.4 mmol), *o*-chloronitrobenzene (6.0 g, 38.1 mmol), and copper-bronze (10.0 g) were heated under reflux at 200° (bath). The cold product was powdered and continuously extracted for 24 h with methylene chloride, and the extract was distilled to give decafluorobiphenyl (2.20 g, 33%), b.p. <158° at 2 mmHg, m.p. 65—67° [from light petroleum (b.p. 40—60°)] (lit.,<sup>18</sup> 68—69°), a fraction of b.p. *ca.* 158° at 2 mmHg, and 2,2'-dinitrobiphenyl (0.32 g, 7%), b.p. >158° at 2 mmHg, m.p. 124—125° [from benzene—light petroleum (b.p. 40—60°)] (lit.,<sup>17</sup> 124°). The middle fraction was recrystallised from light petroleum (b.p. 40—60°) to give yellow diamond-shaped crystals of 2,3,4,5,6-pentafluoro-2'-nitrobiphenyl (1.94 g, 18%) (Found: C, 50.0; H, 1.4; N, 4.6. Calc. for C<sub>12</sub>H<sub>4</sub>F<sub>5</sub>NO<sub>2</sub>: C, 49.9; H, 1.4; N, 4.8%), m.p. 83.0—83.5° (lit.,<sup>7</sup> 84—84.5°); evaporation of the mother liquors yielded impure *o*-chloronitrobenzene (0.52 g, 9%). Use of pentafluoroiodobenzene as the starting material for this synthesis gave a much lower yield of the pentafluoro-2'-nitro-compound.

(b) **2,3,4,5,6-Pentafluoro-3'-nitrobiphenyl.** Bromopentafluorobenzene (5.0 g, 21.2 mmol), *m*-bromonitrobenzene (4.1 g, 20.3 mmol), and copper-bronze (6.4 g) were heated *in vacuo* in a sealed tube at 200° for 48 h. Pentafluorobenzene (*ca.* 0.5 g, 15%) was obtained, and extraction of the solid products with methylene chloride, followed by distillation *in vacuo* and recrystallisation from methylated spirit

<sup>18</sup> L. A. Wall, R. E. Donadio, and W. J. Pummer, *J. Amer. Chem. Soc.*, 1960, **82**, 4846.

gave decafluorobiphenyl (1.10 g, 33%) and 2,3,4,5,6-pentafluoro-3'-nitrobiphenyl (0.72 g, 12%) (Found: C, 49.8; H, 1.4; N, 5.0%) as yellow needles, m.p. 113—114° (lit., 106—108°, <sup>11a</sup> 111—112°<sup>12</sup>).

(c) 2,3,4,5,6-Pentafluoro-4'-nitrobiphenyl. Copper-bronze (7.0 g) was added in small amounts during 30 min to bromopentafluorobenzene (7.0 g, 28.4 mmol) and *p*-iodonitrobenzene (5.0 g, 20.0 mmol), heated at 200° (bath). Heating under reflux was maintained for a further 6 h, the mixture was cooled, and a mixture of bromopentafluorobenzene and pentafluorobenzene (1.41 g), identified by i.r. spectroscopy, was removed under vacuum. The solid product was extracted with methylene chloride, and the extract was distilled to give decafluorobiphenyl (0.67 g, 16%), b.p. < 80° at 2 mmHg, and a fraction, b.p. 140—150° at 2 mmHg, which was recrystallised twice from ethanol to give 2,3,4,5,6-pentafluoro-4'-nitrobiphenyl (1.81 g, 31%) (Found: C, 49.7; H, 1.6; N, 4.9%), m.p. 92—93° (lit., 92°, <sup>7</sup> 87—89°<sup>11a</sup>).

*Nitration of 2,3,4,5,6-Pentafluorobiphenyl.*—The biphenyl (19.7 g, 81.0 mmol) and fuming nitric acid (40 ml; *d* 1.5) were mixed at room temperature and kept for 1 h; the exothermic reaction was then complete. The mixture was poured into cold water, and the products were extracted with methylene chloride; the extract was washed with water, saturated aqueous sodium hydrogen carbonate, and water again, dried (MgSO<sub>4</sub>), and distilled. Distillation of the residue gave the mixed biphenyls, b.p. < 150° at 2 mmHg, which were separated by repeated fractional crystallisation from light petroleum (b.p. 40—60°) and identified by i.r. spectroscopy as the 2'-nitrobiphenyl (4.30 g, 18%), m.p. 83.5—83.8°, the 3'-nitrobiphenyl (1.28 g, 5%), m.p. 112—113°, and the 4'-nitrobiphenyl (11.09 g, 47%), m.p. 88.5—89°. The 2'- and 3'-nitro-isomers tended to crystallise out together, but their different crystalline forms facilitated mechanical separation prior to recrystallisation. Although analytical separation of the nitro-compounds by g.l.c. was readily accomplished, quantitative analysis of the crude mixture gave irreproducible, and therefore unreliable, results.

*Pentafluorophenylation of Nitrobenzene.*—Pentafluorophenylhydrazine (10.0 g, 50.5 mmol) in nitrobenzene (100 ml) was added under nitrogen during 1 h to a stirred suspension of bleaching powder (60.0 g) in nitrobenzene (200 ml) at 90° (bath), and the mixture was kept at 90° for a further 1 h. Nitrogen (800 ml, 71%) was evolved. Insoluble material was removed at the end of the reaction by centrifugation and the excess of nitrobenzene was removed by distillation (17 mmHg). The organic residue was sublimed repeatedly *in vacuo* at 100—110° (bath) and recrystallised from methylated spirit to give 2,3,4,5,6-pentafluoro-3'-nitrobiphenyl (1.40 g, 10%), m.p. 110—111°, identified by i.r. spectroscopy. Treatment of the biphenyl (0.47 g, 1.63 mmol) with fuming nitric acid (5.0 ml; *d* 1.5) under reflux for 24 h gave 3,4-dinitrobenzoic acid (0.25 g, 73%), m.p. and mixed m.p. 161—162° (lit., <sup>17</sup> 163—164°), identified by i.r. spectroscopy.

2,3,5,6-Tetrafluoro-4-hydroxybiphenyl.—2,3,4,5,6-Pentafluorobiphenyl (10.0 g, 40.9 mmol), potassium hydroxide pellets (6.0 g, 107 mmol), and *t*-butyl alcohol (100 ml) were heated under reflux for 49 h. Water (100 ml) was added to the cooled solution and the bulk of the alcohol was distilled off at 80—85°. Extraction of the alkaline solution with methylene chloride yielded no products, but acidification with aqueous 5*M*-hydrochloric acid yielded a white precipitate, which was recrystallised from ethanol to give fine needles of 2,3,5,6-tetrafluoro-4-hydroxybiphenyl (3.8 g,

39%) [Found: C, 59.7; H, 2.6%; *M* (mass spec.), 242. C<sub>12</sub>H<sub>6</sub>F<sub>4</sub>O requires C, 59.5; H, 2.5%; *M*, 242], m.p. 170—171°. Its i.r. and mass spectra were consistent with the proposed structure, and its u.v. spectrum (hexane) showed λ<sub>max</sub> 242 nm (ε 15,000) and λ<sub>min</sub> 216 nm (ε 8300).

2,3,5,6-Tetrafluoro-4-methoxybiphenyl.—(a) From 2,3,4,5,6-pentafluorobiphenyl. The biphenyl (6.0 g, 25 mmol) was heated under reflux for 12 h with sodium methoxide [from sodium (0.7 g, 30 mg atom)] in dry methanol (100 ml). The mixture was poured into water (750 ml), and the precipitate was recrystallised repeatedly from ethanol to give 2,3,5,6-tetrafluoro-4-methoxybiphenyl (3.0 g, 80%) [Found: C, 60.8; H, 3.0%; *M* (mass spec.), 256. C<sub>13</sub>H<sub>8</sub>F<sub>4</sub>O requires C, 60.9; H, 3.1%; *M*, 256], m.p. 63—64°, λ<sub>max</sub> (hexane) 243 nm (ε 16,700), λ<sub>min</sub> 216 nm (ε 9400), giving acceptable i.r. and mass spectra.

(b) From the 4-hydroxybiphenyl. The 4-hydroxybiphenyl (0.50 g, 2.1 mmol) was dissolved in methanolic sodium methoxide [from sodium (0.60 g, 26 mg atom) and dry methanol (10 ml)], and the solution was heated under reflux with iodomethane (2 ml) for 4 h. 2,3,5,6-Tetrafluoro-4-methoxybiphenyl (0.27 g, 51%), m.p. and mixed m.p. 63—64°, was then isolated by extraction with ether.

(c) From 2,3,5,6-tetrafluoro-4-iodoanisole. The anisole (3.54 g, 11.6 mmol) <sup>4</sup> and AnalaR benzene (100 ml) were irradiated in a Pyrex flask with light from a Hanovia 500 W mercury discharge tube. Occasionally, the purple solution was decolourised with aqueous 20% sodium thiosulphate, washed with water (20 ml), dried (MgSO<sub>4</sub>), and replaced. After 21 days, the mixture was decolourised in this manner and dried, and the excess of benzene was carefully removed by distillation. Recrystallisation of the residue from aqueous ethanol gave 2,3,5,6-tetrafluoro-4-methoxybiphenyl (1.62 g, 69%) (Found: C, 59.6; H, 3.5%), identified by mixed m.p. determination and i.r. spectroscopy. Some unchanged tetrafluoroiodoanisole (0.70 g, 20%) was recovered from the mother liquors by dilution with water.

*Reaction of 2,3,5,6-Tetrafluoro-4-methoxybiphenyl with Hydriodic Acid.*—The methoxybiphenyl (2.5 g, 9.8 mmol), redistilled aqueous hydriodic acid (10 ml; 55%), and glacial acetic acid (20 ml) were heated under reflux for 3 h. Most of the acetic acid was distilled off, ether (50 ml) was added to the residual mixture, and the ethereal solution was washed with aqueous 20% sodium disulphite (50 ml) and aqueous *m*-sodium carbonate (50 ml) and then extracted with aqueous 5*M*-sodium hydroxide (50 ml). Acidification of the alkaline solution gave 2,3,5,6-tetrafluoro-4-hydroxybiphenyl (0.03 g), m.p. and mixed m.p. 168—170°, identified by i.r. spectroscopy.

2,3,5,6-Tetrafluoro-4-iodobiphenyl.—2,3,5,6-Tetrafluoro-4-iodophenylhydrazine (15.8 g, 51.6 mmol) <sup>16</sup> in benzene (150 ml) was added dropwise to a well stirred suspension of bleaching powder (75 g) in boiling benzene (200 ml) during 2 h, and the mixture was heated under reflux for a further 1 h. The cooled mixture was filtered, the excess of benzene was evaporated off, and the residue was chromatographed on alumina. Elution with benzene–light petroleum (b.p. 40—60°) gave a product which was recrystallised from ethanol to give 2,3,5,6-tetrafluoro-4-iodobiphenyl (6.9 g, 38%) [Found: C, 41.1; H, 1.6; I, 35.9%; *M* (mass spec.), 352. C<sub>12</sub>H<sub>5</sub>F<sub>4</sub>I requires C, 40.9; H, 1.4; I, 36.1%; *M*, 352], m.p. 106—107°, λ<sub>max</sub> (hexane) 248 nm (ε 18,700), λ<sub>min</sub> 220 nm (ε 5000). Continued elution with benzene–ethanol gave a yellow solid and a brown oil, both of which were unidentified.

*2',3',5',6'-Tetrafluoro-p-terphenyl.*— 2,3,5,6-Tetrafluoro-4-iodobiphenyl (1.00 g, 2.84 mmol) in AnalaR benzene (100 ml), contained in a Pyrex flask, was irradiated with light from a Hanovia 500 W mercury discharge tube for 29 days. The solution was washed with aqueous 20% sodium thio-sulphate and then water and dried ( $\text{MgSO}_4$ ). The benzene was removed by distillation, and recrystallisation of the residue from toluene gave *2',3',5',6'-tetrafluoro-p-terphenyl* (0.51 g, 60%), m.p. and mixed m.p. 254—256° (lit.,<sup>5</sup> m.p. 255—257°), identified by i.r. spectroscopy.

*2',2'',3',3'',5',5'',6',6''-Octafluoro-p-quaterphenyl.*—2,3,5,6-Tetrafluoro-4-iodobiphenyl (2.51 g, 7.10 mmol) and copper-

bronze (10.0 g) were heated *in vacuo* in a sealed tube at 200° for 24 h. Continuous extraction of the product with methylene chloride and with carbon tetrachloride gave the quaterphenyl (1.23 g, 77%) (Found: C, 63.8; H, 2.3. Calc. for  $\text{C}_{24}\text{H}_{10}\text{F}_8$ : C, 64.0; H, 2.2%), m.p. 276—277° (lit.,<sup>19</sup> 279—281°).

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<sup>19</sup> W. L. Respess and C. Tamborski, *J. Organometallic Chem.*, 1970, **22**, 251.

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