

Reactions of Alkenes with Iodine(III) Tris(trifluoroacetate)

Yasuo FUTAMI, Hiroshi NISHINO, and Kazu KUROSAWA*

Department of Chemistry, Faculty of Science, Kumamoto University, Kurokami 2-39-1, Kumamoto 860
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The reactions of 1-arylethene, 1,1-diarylethenes, and 1,1-diarylpropenes with iodine(III) tris(trifluoroacetate) gave 1,2-diaryl-1-alkanones, 1-aryl-2-(4-iodophenyl)-1-alkanones, benzoin, benzils, and iodoethenes. A similar reaction of 1,1,4,4-tetraaryl-1,3-butadiene yielded 1,2,4,4-tetraaryl-3-buten-1-one. The reactions of 1,1,5,5-tetraaryl-1,4-pentadienes and 1,1,6,6-tetraaryl-1,5-hexadienes also gave dicarbonyl compounds. The reaction involves aryl migration. The mechanisms and the utility of the reaction for organic synthesis are discussed.

Among the hypervalent iodine compounds, iodine(III) tris(trifluoroacetate) (ITT) has not been well investigated. This is partly because the reagent is extremely unstable towards moisture, although it is readily prepared. However, several interesting reactions of this reagent with alkanes, alkenes, ethers, and ketones have been reported.¹⁾ During our previous investigation of the reaction of aromatic ketones with ITT²⁾ we found that 1,2-diphenylethanone gave 2-hydroxy-2-(2-iodophenyl)-1-phenylethanone (**5a**) in fairly good yield. This may suggest that iodine could be incorporated into the aromatic ring via an intermediate involving enol form of **5a**. We therefore investigated the reactions of 1,1-diarylethenes, 1,1-diarylpropene, and 1,1, ω,ω -tetraarylalkadienes; however, these gave mainly 1,2-diaryl-1-alkanones and aryl-substituted diketones. It seems to us that such reactions may have a synthetic utility in the preparation of 1,2-diarylethanones and 1,2-diarylpropanones; thus, these reactions were examined by changing the molar ratios of the substrate versus ITT, the substituent in the aromatic ring as well as in olefinic double bond, the reaction temperature, the reaction time, and the solvents.

Results and Discussion

1,1- and 1,2-Diarylethenes. When the reactions of 1,1-diphenylethene (**1a**) with ITT were carried out in various molar ratios at 23 °C for 3 h (Table 1), the products were found to be 1,2-diphenylethanone (**2a**),

1-phenyl-2-(4-iodophenyl)ethanone (**3a**), 2-hydroxy-1,2-diphenylethanone (**4a**), and 2-hydroxy-2-(2-iodophenyl)-1-phenylethanone (**5a**). The best total yield for the products was attained at a molar ratio of 1:1 (Table 1, Entry 3). The yield also depended on the temperature: it gave a similar yield at lower temperature, though the major product was **3a** at the reflux temperature (Entry 4). The products distribution changed greatly, depending on the solvent used (Entries 7–12). The reaction in benzene yielded only two products, **2a** and 2-iodo-1,1-diphenylethene (**6a**). In dichloromethane and hexane the reaction gave almost the same products as in the reaction in CCl₄, but in poor yields. In tetrahydrofuran and acetonitrile **6a** was found to be the sole product in poor yield. The reaction was also examined by changing the reaction times (Entries 13–18). It was observed that the maximum yield of **2a** was attained after 1 h, and then the yield gradually decreased, while **3a** slightly increased.

The reactions were examined for 1-(4-fluorophenyl)-1-phenylethene (**1b**), 1-(4-chlorophenyl)-1-phenylethene (**1c**), 1-(4-bromophenyl)-1-phenylethene (**1d**), 1-(4-chlorophenyl)-1-(4-fluorophenyl)ethene (**1e**), 1,1-bis(4-chlorophenyl)ethene (**1f**), 1-(4-bromophenyl)-1-(4-chlorophenyl)ethene (**1g**), 1-(4-chlorophenyl)-1-(4-iodophenyl)ethene (**1h**), 1,1-bis(4-fluorophenyl)ethene (**1i**), and 1,1-bis(4-methoxyphenyl)ethene (**1j**). The results are shown in Table 2.

1,2-Diarylethanone obtained from **1b** was a mixture of 2-(4-fluorophenyl)-1-phenylethanone (**2b**) and 1-(4-fluorophenyl)-2-phenylethanone (**2'b**) with an isomer ratio of 69:31, which was determined by measuring the intensities of the signals due to the methylene groups in their ¹H NMR spectra with the help of a shift reagent, Eu(fod)₃. 1,2-Diarylethanones from **1d**, **1e**, and **1g** were also found to be a mixture of two isomers, the ratios of which were determined in the same way as mentioned above (Table 2). Compounds **1h–j** gave an ethanone (**2i**), ethanediones (**7h**, **i**, **8j**), and a 1,1-diiodoethene (**9i**). The reaction of *trans*-stilbene (**1k**) yielded *erythro*-1,2-diphenyl-1,2-ethanediol (**10k**) (Entry 28).

Arylpropenes. Aryl migration was also observed in the reaction of arylpropenes as in that of 1,1-

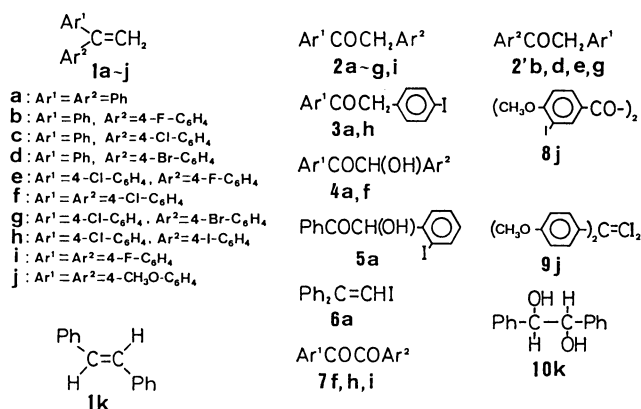
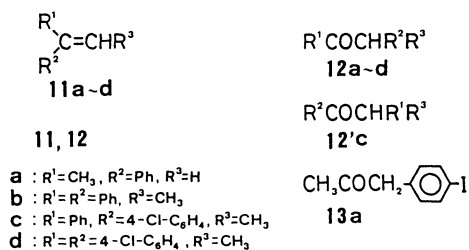


Table 1. Reactions of 1,1-Diphenylethene (**1a**) with I(OCOCF₃)₃

Entry	Molar ratio ^{a)}	Solvent	Temp	Time	Recovery	Product (yield/%) ^{b)}				
			°C	h	%	2a	3a	4a	5a	6a
1	1:0.5	CCl ₄	23	3	27			12		18
2	1:1	CCl ₄	0	3		61	3	4		
3	1:1	CCl ₄	23	3		69	13	9	3	
4	1;1	CCl ₄	reflux	3			32			
5	1:1.5	CCl ₄	23	3			70	5	6	
6	1:2	CCl ₄	23	3			39			
7	1:1	CH ₂ Cl ₂	23	3		22	43	3	22	
8	1:1	CHCl ₃	23	3		39		15		
9	1:1	THF	23	3	32					21
10	1:1	Benzene	23	3	16	53				20
11	1:1	Hexane	23	3		48	29	9		
12	1:1	CH ₃ CN	23	3	28					18
13	1:1	CCl ₄	23	0.25	12			18		10
14	1:1	CCl ₄	23	0.5	5	17		15		6
15	1:1	CCl ₄	23	1		72	9	10	2	
16	1:1	CCl ₄	23	6		69	11	8	2	
17	1:1	CCl ₄	23	12		38	44	7	6	
18	1:1	CCl ₄	23	18		35	45	5	6	

a) Substrate: I(OCOCF₃)₃. b) Yields are based on the substrate added.Table 2. Reactions of Ethenes, Propenes, and Dienes with I(OCOCF₃)₃ at the Molar Ratio of 1:1 in CCl₄ at 23 °C for 3 h

Entry	Substrate	Product (yield/%) ^{a)}				
		Ethanone	2-Hydroxy-ethanone	Ethanedione	Iodoethene	Ethanediol
19	1b	2b,2'b (63) ^{b)}				
20	1c	2c (36) 3h (46)				
21	1d	2d,2'd (64) ^{c)}				
22	1e	2e,2'e (74) ^{d)}				
23	1f	2f (74)	4f (6)	7f (19)		
24	1g	2g,2'g (76) ^{e)}				
25	1h			7h (7)		
26	1i	2i (24)		7i (26)		
27	1j			8j (5)	9j (17)	
28	1k					10k (23)
29	11a	12a (39) 13a (32)				
30	11b	12b (46)				
31	11c	12c,12'c (79) ^{f)}				
32	11d	12d (73)				

a) Yields are based on the substrate added. b) **2b**:**2'b**=69:31. c) **2d**:**2'd**=48:52.d) **2e**:**2'e**=65:35. e) **2g**:**2'g**=50:50. f) **12c**:**12'c**=69:31.respectively (Entries 30—32). The isomer ratio was found to be 69:31 for **12c** and **12'c**.

1,1,ω,ω-Tetraarylalkadienes. We also studied the reactions of 1,1,ω,ω-tetraarylalkadienes. The reactions of 1,1,4,4-tetraphenyl-1,3-butadiene (**14a**) and 1,1,4,4-tetrakis(4-chlorophenyl)-1,3-butadiene (**14b**) with ITT gave allyl phenyl ketones, i.e. 1,2,4,4-tetraphenyl-3-buten-1-one (**15a**) and 1,2,4,4-tetrakis(4-chlorophenyl)-3-buten-1-one (**15b**). The reactions of 1,1,5,5-tetraphenyl-1,4-pentadiene (**14c**) and 1,1,5,5-tetrakis(4-chlorophenyl)-1,4-pentadiene (**14d**) yielded diketones, i.e. 1,2,4,5-tetraphenyl-1,5-pentanedione (**16c**) and 1,2,4,5-tetrakis(4-chlorophenyl)-1,5-pentanedione (**16d**). Similarly, the reaction of 1,1,6,6-tetraphenyl-1,5-hexadiene (**14e**) and 1,1,6,6-tetrakis(4-

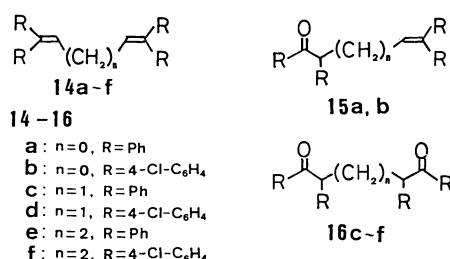
diarylethenes. 2-Phenylpropene (**11a**) gave 1-phenyl-2-propanone (**12a**) and 1-(4-iodophenyl)-2-propanone (**13a**) (Entry 29). 1,1-Diphenylpropene (**11b**), 1-(4-chlorophenyl)-1-phenylpropene (**11c**), and 1,1-bis(4-chlorophenyl)propene (**11d**) yielded the corresponding 1,2-diaryl-1-propanones, (**12b**, **12c**, **12'c**, and **12d**),

Table 3. Reactions of Dienes (**14a–f**) with ITT at a Molar Ratio of 1 : 1 at 23 °C for 3 h

Entry	Substrate	Product (Yield/%) ^{a)}	
		Aryl ketone	Diketone
32	14a	15a (37) ^{b)}	
33	14b	15b (23)	
34	14c		16c (10)
35	14d		16d (44)
36	14e		16e (16)
37	14f		16f (77)

a) Yields are based on the substrate added. b) Substrate was recovered (60%).

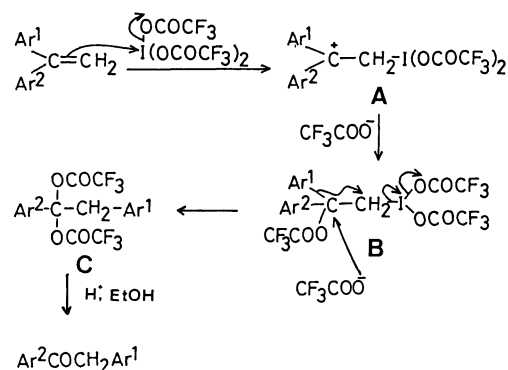
chlorophenyl)-1,5-hexadiene (**14f**) gave 1,2,5,6-tetraphenyl-1,6-hexanedione (**16e**) and 1,2,5,6-tetrakis(4-chlorophenyl)-1,6-hexanedione (**16f**), respectively (Table 3). These diketones (**16c–f**) can be a mixture



of two diastereoisomers; however, the ^1H NMR spectra of these compounds showed that only one of the isomers is produced. The reaction of 1,1,2-triphenylethene with ITT yielded a complex mixture, the products of which could not be characterized.

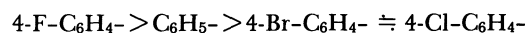
Reaction Mechanisms. The reaction of alkenes with ITT has been reported by Buddrus,³⁾ who described that trifluoroacetoxyl groups added to the double bond to form mainly 1,2-glycol bis(trifluoroacetate). The reaction of styrene was briefly mentioned as giving 1,1-bis(trifluoroacetate) as well as 1,2-bis(trifluoroacetate).⁴⁾ Our study revealed that 1,1-diaryl-1-alkenes always gives ketones. The reaction mechanism could be depicted as involving 1,2-aryl shift (**B**→**C**) and the hydrolysis of the reaction intermediate (**C**) upon treatment with aqueous acid (Scheme 1). The ^1H NMR (CCl_4) spectrum of the reaction mixture after an hour of stirring showed a singlet at $\delta=3.83$ corresponding to the methylene group in **C**. The IR spectrum (CCl_4) also exhibited a strong absorption at 1802 cm^{-1} due to a trifluoroacetoxyl group. Evidence for an intramolecular rearrangement was obtained when a mixed substrate, **1a** and **1f**, was subjected to a reaction with ITT. The only products that we could find were **2a**, **2f**, and **3a**; there was no crossover product, such as 2-(4-chlorophenyl)-1-phenylethanone and 1-(4-chlorophenyl)-2-phenylethanone.

The substituent in the aromatic ring has a significant effect on the migratory aptitude of the aryl group. As shown in the isomer ratio of **2b**:**2'b**, which is in



Scheme 1.

favor of **2b**, the 4-fluorophenyl group is more movable than the phenyl group. The phenyl group, on the other hand, is more readily transferred than the 4-bromophenyl and 4-chlorophenyl groups, the latter two being about the same in migratory aptitude. Thus, the order of migratory aptitude is as follows:



1-Aryl-2-(4-iodophenyl)ethanones (**3a** and **3h**) and 1-(4-iodophenyl)-2-propanone (**13a**) may be formed after a migration of the phenyl group in intermediate **A**. The formation of 2-hydroxyethanone (**5a**) and ethanediones (**7**) has already been discussed in a previous paper.²⁾

The formation of iodoethenes, **6a** and **9j**, indicates the electron-donating nature of phenyl and 4-methoxyphenyl groups. These groups stabilize the carbonium ion **A** which is deprotonated and, eventually, affords the iodoethenes (**6a** and **9j**).

In conclusion, the reaction of ITT proved to be useful for the preparation of ethanones and 1,1, ω , ω -tetraarylalkanediones from 1,1-diarylethenes and 1,1, ω , ω -tetraarylalkadienes, respectively. The reaction involves intramolecular aryl migration, confirmed by the absence of the crossover products in the product mixture.

Experimental

Measurements. All ^1H NMR spectra were taken in deuteriochloroform or carbon tetrachloride solutions with tetramethylsilane as the internal standard on a JNM-PMX 60 SI spectrometer (60 MHz). The IR spectra were measured in chloroform solutions on a JASCO A-102 IR spectrometer. The mass spectra were obtained with JMS-OISG-2 and JMS-DX-303 HF instruments using a direct-insertion probe at an ionizing voltage of 70 eV. The melting points were determined with a Yanagimoto micromelting point apparatus and were not corrected.

Materials. Arylethenes (**1a–j**) arylpropenes (**11a–d**) and 1,1, ω , ω -tetraarylalkadienes (**14a–f**) were obtained by the dehydration of the corresponding alcohols, which were prepared by a Grignard reaction.⁵⁾ **1k** was commercially available from Katayama Chemical Industries. **1g**: Mp 71–72 °C (ethanol); IR ν (cm^{-1}) 1659 (C=C); ^1H NMR

(CDCl₃) δ =5.51 (2H, s, =CH₂), 7.03–7.30 (6H, m, ar.H), and 7.34–7.59 (2H, m, H-2', H-6'). Found: m/z 291.9653, Calcd for C₁₄H₁₀⁷⁹Br³⁵Cl: M, 291.9655. **1h**: Mp 67 °C (ethanol); IR ν (cm⁻¹) 1660 (C=C); ¹H NMR (CDCl₃) δ =5.41 (2H, s, =CH₂), 6.89–7.16 (2H, m, H-3', H-5'), 7.19–7.46 (4H, m, ar.H), and 7.49–7.76 (2H, m, H-2', H-6'). Found: m/z 339.9542, Calcd for C₁₄H₁₀³⁵ClI: M, 339.9516.

Reactions of Iodine(III) Tris(trifluoroacetate). The general procedure for the reaction of ITT was as follows. To a solution of a substrate (1 or 2 mmol) in a solvent (20 cm³), iodine(III) tris(trifluoroacetate)²⁾ (0.5–2 mmol, as shown in Tables 1, 2, and 3), was added under a nitrogen atmosphere. The solution was stirred with undissolved ITT at 23 °C for 3 h. Water (30 cm³) was added to the reaction mixture and the mixture was stirred again for 1 h. The reaction mixture was then extracted with chloroform and the organic layer was separated and washed with 5% aqueous sodium thiosulfonate solution, and water. The solvent was removed under reduced pressure and the residue was heated with a mixture of ethanol (30 cm³) and concd hydrochloric acid (0.5 cm³) at 100 °C for 1 h. The solvent was removed under reduced pressure. The products were separated on TLC with benzene as the developing solvent. The yields on reasonably pure products are summarized in Tables 1, 2, and 3. Analytical samples were further purified by recrystallization. The known compounds were identified by comparisons of their spectral data and/or the melting points with those found in the literature.

Reaction Products with ITT. **2a**: Mp 53–54 °C (lit.⁶) mp 55–56 °C). Mixture of **2b** and **2'b**: **2b**:**2'b**=69:31; mp 91–92 °C (lit.⁷) mp 86 °C for **2b**, lit.⁸) mp 11 °C for **2'b**). **2c**: Mp 135 °C (lit.⁹) mp 135.5–136.5 °C). Mixture of **2d** and **2d'**: **2d**:**2d'**=48:52; mp 132–133 °C (lit.¹⁰) mp 146–147 °C for **2d**, lit.¹¹) mp 104–105 °C for **2d'**). Mixture of **2e** and **2e'**: **2e**:**2e'**=65:35; mp 85.5–86.5 °C (ethanol); IR ν (cm⁻¹) 1680 (>C=O); ¹H NMR (CDCl₃) δ =4.44 (4H, s, -CH₂-, -CH₂-), 6.88–7.49 (12H, m, ar.H), and 7.83–8.24 (4H, m, H-2', H-2', H-6', H-6'). Found: m/z 248.0397, Calcd for C₁₄H₁₀³⁵ClFO: M, 248.0404. **2f**: Mp 113–114 °C (lit.¹²) mp 112.5–113.5 °C). Mixture of **2g** and **2g'**: **2g**:**2g'**=50:50; mp 123–124 °C (lit.¹³) mp 121–121.5 °C for **2g**, lit.¹³) mp 106–107 °C for **2g'**). **2i**: Mp 96.5–98 °C (lit.¹⁴) mp 96–97 °C). **3a**: Mp 158–159 °C (ethanol); IR ν (cm⁻¹) 1678 (>C=O); ¹H NMR (CDCl₃) δ =4.23 (2H, s, -CH₂-), 6.94–7.74 (7H, m, ar.H), and 7.93–8.14 (2H, m, H-2', H-6'). Found: m/z 321.9885, Calcd for C₁₄H₁₁IO: M, 321.9945. **3h**: Mp 139–140 °C (ethanol); IR ν (cm⁻¹) 1679 (>C=O); ¹H NMR (CDCl₃) δ =4.21 (2H, s, -CH₂-), 6.93–7.07 (2H, m, H-3', H-5'), 7.37–7.51 (2H, m, H-3', H-5'), 7.60–7.73 (2H, m, H-2'', H-6''), and 7.87–8.01 (2H, m, H-2', H-6'). Found: m/z 355.9479, Calcd for C₁₄H₁₀O³⁵ClI: M, 355.9464. **4a**: Mp 133–134 °C (lit.¹⁵) mp 133 °C). **4f**: Mp 87 °C (lit.¹⁶) mp 87–88 °C). **5a**: Mp 114–115 °C (lit.²) mp 113.2–114.2 °C). **6a**: Liquid (lit.¹⁷) mp 40–41 °C); ¹H NMR (CDCl₃) δ =6.91 (1H, s, =CHI), and 7.04–7.47 (10H, m, ar.H). Found: m/z 305.9913, Calcd for C₁₄H₁₁I: M, 305.9910. **7f**: Mp 198 °C (lit.¹⁸) mp 199 °C). **7h**: Mp 210–211 °C (ethanol); IR ν (cm⁻¹) 1674 (>C=O); ¹H NMR (CDCl₃) δ =7.35–7.77 (4H, m, H-3', H-3'', H-5', H-5''), and 7.77–8.07 (4H, m, H-2', H-2'', H-6', H-6''). Found: m/z 369.9283, Calcd for C₁₄H₈³⁵ClIO: M, 369.9258. **7i**: Mp 118 °C (lit.¹⁹) mp 120–121 °C). **8j**: Mp 218–219 °C (ethanol); IR ν (cm⁻¹) 1660 (>C=O); ¹H NMR (CDCl₃) δ =3.96 (6H, s, 2×OCH₃), 6.84 (2H, d, J =8.4 Hz, H-

5', H-5''), 7.93 (2H, dd, J =8.4, 2.0 Hz, H-6', H-6''), and 8.40 (2H, d, J =2.0 Hz, H-2', H-2''). Found: m/z 521.8837, Calcd for C₁₆H₁₂I₂O₄: M, 521.8826. **9j**: Mp 139–140 °C; ¹H NMR (CDCl₃) δ =3.76 (6H, s, 2×OCH₃), 6.67–6.95 (4H, m, H-3', H-3'', H-5', H-5''), and 7.04–7.31 (4H, m, H-2', H-2'', H-6', H-6''). Found: m/z 491.9110, Calcd for C₁₆H₁₄I₂O₂: M, 491.9084. **10k**: Mp 123–124 °C (lit.²⁰) mp 119–120 °C). **12a**: Liquid (lit.²¹) bp 97–98.5 °C/ 1.73×10³ Pa). **12b**: Liquid (lit.²²) bp 63–70 °C/ 0.67 Pa). Mixture of **12c** and **12c'**: Liquid; **12c**:**12c'**=69:31; IR ν (cm⁻¹) 1680 (>C=O); ¹H NMR (CDCl₃) δ =1.44 (6H, d, J =7.2 Hz, CH₃, CH₃), 4.58 (2H, q, J =7.2 Hz, 2×>CH-), 6.80–7.65 (14H, m, ar.H), and 7.65–8.05 (4H, m, H-2', H-2', H-6', H-6'). Found: m/z 244.0659, Calcd for C₁₅H₁₈³⁵ClO: M, 244.0655. **12d**: Mp 62.5–63.5 °C (ethanol); IR ν (cm⁻¹) 1682 (>C=O); ¹H NMR (CCl₄) δ =1.45 (3H, d, J =6.6 Hz, CH₃), 4.51 (1H, q, J =6.6 Hz, >CH-), 6.95–7.48 (6H, m, ar.H), and 7.62–7.98 (2H, m, H-2', H-6'). Found: m/z 278.0250, Calcd for C₁₅H₁₂³⁵Cl₂O: M, 278.0265. **13a**: Liquid (lit.²³) bp 135–136 °C/266 Pa). **15a**: Liquid (lit.²⁴) mp 91.5–93 °C); IR ν (cm⁻¹) 1678 (>C=O); ¹H NMR (CDCl₃) δ =5.30 (1H, d, J =10.2 Hz, -COCH<), 6.66 (1H, d, J =10.2 Hz, =CH-), 6.93–7.55 (18H, m, ar.H), 7.55–7.83 (2H, m, H-2', H-6'). **15b**: Liquid; IR ν (cm⁻¹) 1680 (>C=O); ¹H NMR (CCl₄) δ =5.15 (1H, d, J =10.2 Hz, -COCH<), 6.55 (1H, d, J =10.2 Hz, =CH-), 6.87–7.48 (14H, m, ar.H), and 7.48–7.77 (2H, m, H-2', H-6'). Found: m/z 510.0083, Calcd for C₂₈H₁₈³⁵Cl₄O: M, 510.0112. **16c**:²⁵) Liquid; IR ν (cm⁻¹) 1679 (>C=O); ¹H NMR (CDCl₃) δ =2.00–3.12 (2H, m, -CH₂-), 4.17–4.74 (2H, m, 2× -COCH<), 6.71–7.57 (16H, m, ar.H), 7.57–8.00 (4H, m, H-2', H-2', H-6', H-6'). **16d**: Mp 164–165 °C (ethanol); IR ν (cm⁻¹) 1679 (>C=O); ¹H NMR (CCl₄) δ =1.97–3.03 (2H, m, -CH₂-), 4.13–4.63 (2H, m, 2× -COCH<), 6.87–7.47 (12H, m, ar.H), and 7.53–7.93 (4H, m, H-2', H-2', H-6', H-6'). Found: m/z 540.0189, Calcd for C₂₉H₂₀³⁵Cl₄O₂: M, 540.0217. **16e**:²⁶) Mp 186–187 °C (ethanol); IR ν (cm⁻¹) 1678 (>C=O); ¹H NMR (CDCl₃) δ =1.53–2.40 (4H, m, -CH₂CH₂-), 4.27–4.77 (2H, m, 2× -COCH<), 7.10–7.53 (16H, m, ar.H), 7.76–8.03 (4H, m, H-2', H-2', H-6', H-6'); MS m/z (rel intensity)=418 (M⁺, 6), 296 (75), 117 (100), 105 (100), 91 (48), 77 (60). **16f**: Mp 159–161 °C (ethanol); IR ν (cm⁻¹) 1679 (>C=O); ¹H NMR (CDCl₃) δ =1.39–2.36 (4H, m, -CH₂CH₂-), 4.12–4.60 (2H, m, 2× COCH<), 6.86–7.43 (12H, m, ar.H), and 7.56–7.88 (4H, m, H-2', H-6'). Found: m/z 554.0388, Calcd for C₃₀H₂₂³⁵Cl₄O₂: M, 554.0374.

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