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(2+2) Photosensitized cycloadditions of maleic anhydride derivatives to selenophene and tellurophene derivatives

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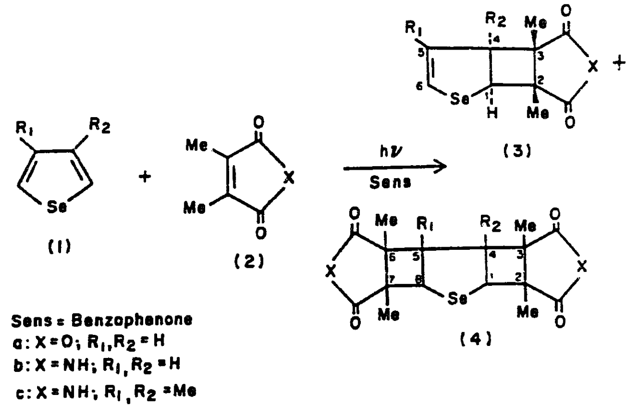
Abstract

The photoadditions of 2,3-dimethylmaleic anhydride to selenophene (C. Rivas, D. Pacheco, F. Vargas, J. Heterocycl. Chem. 17 (1980) 1151) and selenophthene (D. Pacheco, C. Rivas, F. Vargas, J. Heterocycl. Chem. 20 (1983) 1465) and of 2,3-dimethylmaleimide to selenophene and 3,4-dimethylselenophene (C. Rivas, F. Vargas, A. Torrealba, D. Pacheco, R. Machado, G. Aguiar, Monatsh. Chem. 125 (1994) 1153) in benzophenone-photosensitized reactions have been carried out successfully in this laboratory. This reaction has been extended to other methyl-substituted selenophenes and, during the course of this investigation, it was observed that the compounds under study showed a somewhat different behaviour from those mentioned above. The new photoproducts obtained are reported and an attempt is made to give a general view of the photochemical behaviour of methyl selenophenes. Furthermore, tellurophene and benzo(b)tellurophene were also tested as substrates for these (2+2) cycloaddition reactions. © 1997 Elsevier Science S.A.

Keywords: Maleic anhydride; Maleimide; Oxetane benzophenone; Photocycloaddition; Selenophene; Selenophthene; Tellurophene

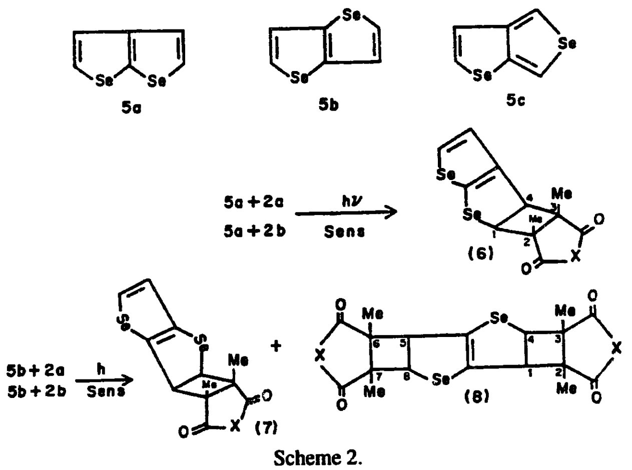


Scheme 1.

1. Introduction

Derivatives of the common five-membered heterocycles furan [1], thiophene [2], pyrrole [3] and, to a lesser extent, selenophene [4] and tellurophene have been used as substrates in (2+2) photoaddition reactions with carbonyl compounds to give oxetanes [5] and with compounds containing carbon–carbon double bonds, such as maleic anhydride derivatives, to yield cyclobutane-containing compounds. The photosensitized additions of 2,3-dimethylmaleic anhydride and its nitrogen analogues (imides) to selenophene derivatives [4,6] and selenophthene [7] are illustrated in Schemes 1 and 2 respectively.

In order to obtain a general view of the photochemical behaviour of selenophenes and selenophthenes as substrates for the maleimides, in addition to selenophene and 3,4-dimethylselenophene which have been studied previously [6], 2-methylselenophene, 3-methylselenophene, 2,5-dimethylselenophene and selenophthene (cis and trans) were tested



Scheme 2.

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as substrates for 2,3-dimethylmaleimide. Furthermore, 2,3-dimethylmaleimide, *N*-methylmaleimide and *N*-phenylmaleimide were used as possible photoadducts to 3,4-dimethylselenophene.

Citraconic anhydride, which was considered to be inert to this type of reaction on the basis of preliminary results, added to selenophene in a less efficient reaction than that of 2,3-dimethylmaleic anhydride. Of the tellurophene series, only tellurophene and benzo(b)tellurophene were tested as substrates for the methyl-substituted maleimides.

All of these reactions were sensitized by benzophenone, and structural elucidation of the compounds was carried out by spectroscopic methods, in particular proton nuclear magnetic resonance (^{1}H NMR), ^{13}C NMR, mass spectrometry and elemental analysis.

2. Experimental details

Melting points were measured with a Fischer–Johnes apparatus and were uncorrected. IR spectra were obtained with an FT-IR Nicolet DX V 5.07 spectrometer. All the ^{1}H NMR, ^{13}C NMR and two-dimensional H, C-correlated (COSY) spectra were recorded on a Brucker Aspect 3000 (300 MHz) instrument. Tetramethylsilane (TMS) was used as internal standard and the solvents employed were deuterated chloroform and deuterated dimethylsulphoxide. The mass spectra were determined in a Carlo-Erba/Kratos MS25RFA spectrometer coupled to a gas chromatograph with a capillary column (25 m, 5% phenylmethylsilicone).

Irradiations were carried out with a Rayonet photochemical reactor equipped with 12 low pressure mercury lamps (250–350 nm), a Normag photoreactor equipped with a medium pressure mercury arc and a Hanovia 450 W mercury arc.

Elemental analyses were carried out in the Elemental Analysis Laboratory of the Venezuelan Institute of Scientific Research using a Carlo-Erba/Kratos MS25RFA mass spectrometer.

Benzophenone, 2,3-dimethylmaleic anhydride and citraconic anhydride were purchased from Aldrich; the latter was carefully distilled before use. Selenophene was prepared according to the method of Gronowitz and Frejd [8] modified by Vargas. 2-Methylselenophene [9], 3-methylselenophene [10], 2,5-dimethylselenophene [10] and 3,4-dimethylselenophene [10] were prepared by the methods reported in the literature. The selenophthenes, cis (**5a**) and trans (**5b**), were isolated as byproducts in the synthesis of selenophene; the iso form (**5c**) could not be isolated [11–13]. 2,3-Dimethylmaleimide was prepared by the treatment of 2,3-dimethylmaleic anhydride with ammonia [14]. Benzo(b)tellurophene was prepared by the method of Bradsma et al. [15].

2.1. Photochemical reactions

A typical experiment serves to illustrate the general method applied in most reactions. Thus equimolar quantities of the heterocycle (0.01 mol) and 2,3-dimethylmaleic anhydride or amide (0.01 mol), in addition to 3.6 g of benzophenone (0.02 mol), were dissolved in 250 ml of dry benzene and irradiated with a Hanovia 450 W medium pressure lamp for 8 h at 20 °C under a nitrogen atmosphere in a quartz immersion photoreactor (Applied Photophysics Part No. 3230+3307) fitted with a Pyrex filter sleeve. In most of the experiments, after about 3 h of exposure to UV light, small crystals began to appear on the walls of the irradiation apparatus. At the end of irradiation, the crystals were filtered and washed with petroleum ether. Separation of the products and further purification were performed by column chromatography on neutral alumina. The column was eluted with benzene–acetone (3 : 1). The cycloaddition reactions can also be carried out in a Rayonet reactor. When reactions were carried out with very small amounts of reagents, a Normag photoreactor (125 W mercury lamp) was used.

After work-up, two products were isolated whose structures were elucidated as follows.

2.2. Photocycloaddition products

2.2.1. (2+2) Photoadducts of selenophene

2.2.1.1. Adduct **11a** (N-methylmaleimide + 3,4-dimethylselenophene)

Yield 70% (acetone–petroleum ether, 1 : 1); m.p. > 250 °C (decomp.). IR (KBr pellet) ν: 1700–1686 (C=O), 2961, 1261 (CH$_3$), 803 (CH). ^{1}H NMR (DMSO-d_6) δ: 4.79 (s, 2H, 1-H and 8-H), 2.73 (s, 2H, 2-H and 7-H), 3.11 (s, 3H, N-CH$_3$), 2.91 (s, 2H, 3-H and 6-H), 1.66 (s, 6H, 4-CH$_3$ and 5-CH$_3$). ^{13}C NMR (DMSO-d_6) δ: 177.8 (C=O), 42.09 (C-1 and C-8), 41.17 (C-2 and C-7), 34.86 (C-3 and C-6), 39.98 (C-4 and C-5), 24.73 (CH$_3$). MS *m/z* (%): 300 (60), 160 (10), 112 (10), 78 (100).

2.2.1.2. Adduct **11b** (N-phenylmaleimide + 3,4-dimethylselenophene)

Yield 60% (acetone–petroleum ether, 1 : 1); m.p., 126–128 °C. IR (KBr pellet) ν: 2940–2926 (C–H), 1792–1717 (C=O), 1561–1517 (Ph), 1374 (CH$_3$). ^{1}H NMR (DMSO-d_6) δ: 7.46–7.37 (m, 5H, aromatic H), 4.44 (s, 2H, 1-H and 8-H), 3.97 (s, 2H, 2-H and 7-H), 3.67 (m, 2H, 3-H and 6-H), 1.61 (s, 6H, 4-CH$_3$ and 5-CH$_3$). ^{13}C NMR (DMSO-d_6) δ: 175.93 and 169.30 (C=O), 43.33 (C-1 and C-8), 40.2 (C-2 and C-7), 30.0 (C-3 and C-6), 17.56 (CH$_3$-4 and CH$_3$-9), 129, 127 and 126 (Ph). MS *m/z* (%): 506 (M$^+$, 10), 426 (M$^+$ − C$_6$H$_5^+$, 25), 173 (C$_{10}$H$_7$O$_2$N$^+$, 100), 160 (SeC$_6$H$_8^+$, 40), 93 (C$_4$H$_2$O$_2$N$^+$, 100), 73 (C$_6$H$_5^+$, 70).

2.2.1.3. Adduct **12** (2,3-dimethylmaleimide + 2-methylselenophene)

Yield 75%; m.p., 250–252 °C. IR (KBr pellet) ν: 3214 (NH), 1700–1710 (C=O), 1367–1316 (CH$_3$), 1146–1126 (C–O), 804 (CH). ^{1}H NMR (CDCl$_3$) δ: 7.26 (s, 1H, 5-H), 6.40 (s, 1H, 1-H), 4.73 (s, 1H, 4-H), 1.96 (s, 3H, 6-CH$_3$), 1.34 (s, 3H, 2-CH_3), 1.19 (s, 3H, 3-CH_3). ^{13}C NMR ($CDCl_3$) δ: 171.82 (C=O), 138.37 (C-6), 120.51 (C-5), 57.88 (C-2), 49.68 (C-3), 30.0 (C-4), 18.62 (CH_3-3), 14.85 (CH_3-6), 11.70 (CH_3-2). MS *m/z* (%): 257 (M^+, 2), 146 ($C_5H_6Se^+$, 100), 125 ($C_6H_7O_2N^+$, 57), 79 (Se^+, 60), 39 ($C_3H_3^+$, 20).

2.2.1.4. Dimer 13

^1H NMR ($CDCl_3$) δ: 11.46 (s, 1H, NH), 1.05 (s, CH_3). MS *m/z* (%): 250 (M^+, 10), 235 ($M^+ - CH_3$, 15), 125 ($C_6H_7O_2N^+$, 100).

2.2.1.5. Adduct 14

Yield 40%; m.p., 195–196 °C. ^1H NMR (DMSO-d_6) δ: 6.90 (dd, $J_{5,6}=6$ Hz, $J_{1,6}=1.2$ Hz, 1H, 6-H), 5.95 (dd, $J_{5,6}=6$ Hz, $J_{5,4}=2.6$ Hz, 1H, 5-H), 5.2 (dd, $J_{1,2}=7$ Hz, $J_{1,4}=6.5$ Hz, 1H, 1-H), 4.6 (dm, $J_{4,1}=6.5$ Hz, $J_{4,2}=1.2$ Hz, $J_{4,5}=2.6$ Hz, 1H, 4-H), 3.1 (dd, $J_{2,1}=7$ Hz, $J_{2,4}=1.2$ Hz, 1H, 2-H), 1.3 (s 3H, CH_3). ^{13}C NMR (DMSO-d_6) δ: 175, 177 (C=O), 126.47 (C-3), 121.7 (C-4), 78.7 (C-1), 58.26 (C-6), 56.0 (C-7), 54.7 (C-5), 19.5 (CH_3-6). MS (diacid) *m/z* (%): 262 (M^+, 3), 244 ($M^+ - 18$, anhydride, 5), 216 (anhydride − C=O, 5), 132 ($C_4H_4Se^+$, 100), 112 ($C_4H_4O_3^+$, 10), 68 (15), 57 (10).

2.2.2. (2 + 2) Photoadducts of selenophthene

2.2.2.1. Adduct 6

Yield 60%; m.p., 164–165 °C. IR (KBr pellet) ν: 3300 (NH), 1800 (C=O), 1320 (CH_3). ^1H NMR (DMSO-d_6) δ: 8.50 (s, 1H, NH), 8.20–7.50 (2d, $J_{6,7}=5.0$ Hz, 2H, 6-H and 7-H), 5.35 (d, $J_{1,4}=7.0$ Hz, 1H, 1-H), 4.60 (d, $J_{4,1}=7.0$ Hz, 1H, 4-H), 1.30 (s, 3H, 2-CH_3), 1.00 (s, 3H, 3-CH_3). ^{13}C NMR (DMSO-d_6) δ: 180–179 (C=O), 138.5–120.6 (4s, C-aromatic), 47.2 (C-1), 30 (C-4), 13.20 (CH_3-2), 12.9 (CH_3-3). MS *m/z* (%): 361 (M^+, 2), 236 ($C_6H_4Se_2^+$, 47), 125 ($C_6H_7O_2N^+$, 100).

2.2.2.2. Adduct 7

Yield 30%; m.p., 300 °C (decomp.). IR (KBr pellet) ν: 3280 (NH), 1800 (C=O), 1320 (CH_3). ^1H NMR (DMSO-d_6) δ: 7.30 (s, 1H, NH), 3.60 (2d, $J_{1,4}=6.5$ Hz, $J_{5,8}=6.5$ Hz, 2H, 4-H and 8-H), 3.20 (2d, $J_{4,1}=6.5$ Hz, $J_{8,5}=6.5$ Hz, 2H, 1-H and 5-H), 1.50–1.00 (4s, 12H, CH_3s). ^{13}C NMR (DMSO-d_6) δ: 183.3, 182.7, 181.7, 179.6 (C=O), 52.2 (non-carbonyl carbon atoms), 49.5, 48.5, 48.2, 47.2, 38.4, 34.8, 34.6, 34.2 (ring carbon atoms), 16.5, 14.6, 13.2, 12.9 (CH_3s). MS *m/z* (%): 486 (M^+, 2), 236 ($C_6H_4Se_2^+$, 10), 125 ($C_6H_7O_2N^+$, 100).

2.2.3. Oxetanes

2.2.3.1. Oxetane 16

Yield 30%; m.p., 60–62 °C. ^1H NMR ($CDCl_3$) δ: 2.2 (s, 3H, CH_3), 8.0–7.6 (m, Ph), 7.4 (s, 1H, 1-H), 7.2 (d, $J_{3,4}=6.0$ Hz, 1H, 3-H), 6.9 (d, $J_{4,3}=6.0$ Hz, 1H, 4-H). MS *m/z* (%): 311 (M^+, 2), 182 ($C_{13}H_{10}O^+$ (benzophenone), 50), 146 ($C_5H_7Se^+$ (methylselenophene), 41), 105 ($PhCO^+$, 100), 77 ($C_6H_5^+$, 60).

2.2.3.2. Oxetane 17

Yield 60%; m.p., 140–141 °C. ^1H NMR ($CDCl_3$) δ: 7.6 (m, Ph), 5.6, 5.5 (s, s, two protons in the ring), 1.6 (s, 3H, CH_3), 1.5 (s, 3H, CH_3). MS (mixture of **7** and **8**) *m/z* (%): 522 (M^+, 1), 182 ($C_{13}H_{10}O^+$ (benzophenone), 30), 160 ($C_6H_8Se^+$, 100), 105 ($PhCO^+$, 100), 77 ($C_6H_5^+$, 80).

2.2.3.3. Oxetane 18

Yield 40%; m.p., 132–134 °C. ^1H NMR ($CDCl_3$) δ: 7.50–7.30 (m, Ph), 3.70 (s, two protons in the ring), 2.50 (s, 3H, CH_3), 2.42 (s, 3H, CH_3).

2.2.3.4. Oxetane 19

Yield 30%; m.p., 180–181 °C. ^1H NMR (C_2d_6CO) δ: 8.74 (d, $J_{1,5}=8.0$ Hz, 1H, 1-H), 7.90–7.80 (d, $J_{5,1}=8.0$ Hz, 1H, 5-H), 7.70–7.50 (m, Ph). MS *m/z* (%): 232 ($C_8H_6Te^+$, 100), 182 ($C_{13}H_{10}O^+$, 5), 128 (Te, 9), 102 ($C_8H_6^+$, 100). Isotopic distribution of tellurium observed.

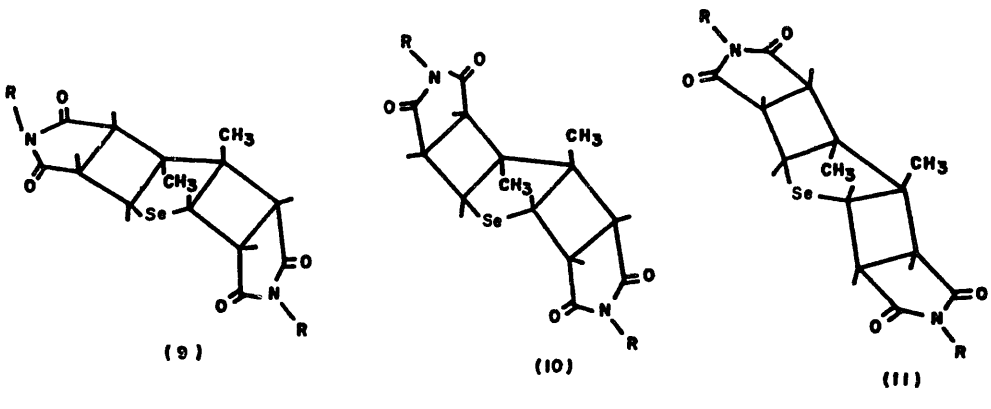
3. Discussion

As observed previously, when 2,3-dimethylmaleimide is irradiated in the presence of selenophene or 3,4-dimethylselenophene in benzophenone-photosensitized reactions, one single (1 : 1) (**3b** and **3c**) and one double (2 : 1) (two molecules of maleimide to one molecule of selenophene) (**4b** and **4c**) adduct is formed, as obtained for 2,3-dimethylmaleic anhydride. Inspection and analysis of the ^{13}C NMR spectra of the adducts leads to the conclusion that selenophene yields one single adduct (1 : 1) and one double adduct (2 : 1), which consists of a mixture of three geometrical isomers (structures given in Fig. 1), whereas 3,4-dimethylselenophene yields one single adduct (1 : 1) but, in contrast with selenophene, only one geometrical isomer of the (2 : 1)-type adduct which is probably the anti anti structure **11a** (Fig. 1) [6]. When models of this compound are built, steric crowding of the methyl groups in positions 3 and 4 of the heterocyclic nucleus prevents the formation of the other two isomers. The same observation is made when *N*-methylmaleimide and *N*-phenylmaleimide are used as substrates for 3,4-dimethylselenophene (structures **11a** and **11b**).

In the present investigation, it was observed that 3-methylselenophene under the same conditions yields the dimer of the maleimide as the only identifiable photoproduct; it is possible that other products in a polymeric residue may be present.

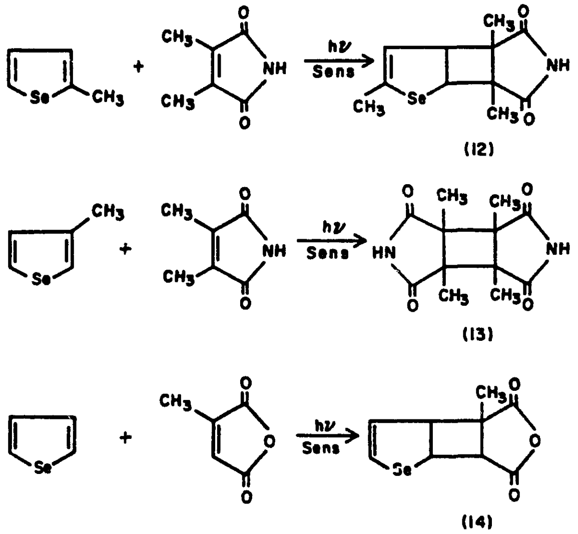
2-Methylselenophene in the presence of 2,3-dimethylmaleimide and benzophenone after irradiation affords one single (1 : 1) adduct (structure **12**) (Scheme 3).

On the other hand, 2-methylselenophene and 3-methylselenophene, irradiated in the presence of benzophenone alone,



R = (a), CH_3, (b) Ph

Fig. 1. Three geometrical isomers of 2 : 1 adduct of selenophene.



Scheme 3.

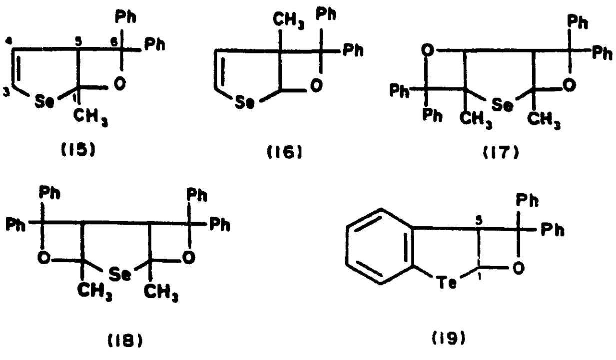


Fig. 2. Oxetanes 15–19.

undergo a Paterno–Büchi reaction to produce oxetanes **15** [8] and **16** respectively. Nevertheless, thus far it has not been possible to isolate the oxetanes when the maleimide is present in the reaction medium (Fig. 2). Furthermore, 2,5-dimethylselenophene under the same reaction conditions cycloadds to the benzophenone to form oxetanes **17** and **18**.

The two oxetanes formed from this heterocycle are 2 : 1 products (two molecules of benzophenone to one molecule of heterocycle to yield a double oxetane) as may be inferred from spectroscopic data. If a single oxetane was present, the protons in the ring would be multiplets (quartets) because of the allylic and homoallylic coupling respectively with the methyl group at position 3. However, compound **17** exhibits two singlets corresponding to the two protons in the ring, and compound **18**, apart from the aromatic resonances, exhibits two singlets, one at 3.69 ppm and the other at 2.49 ppm, which can only be assigned to the two protons in the ring and the two methyl groups respectively as expected for a symmetrical molecule.

It is interesting to note that 3,4-dimethylselenophene, which undergoes the (2+2) cycloaddition reaction in the presence of the maleimides and benzophenone, does not yield oxetane when it is irradiated in the presence of benzophenone alone.

In contrast with thiophene, which gives linear Stern–Volmer plots [11–13] (quencher of excited benzophenone) and allows the quenching constants to be calculated, thus giving a clear indication of the quenching ability of these compounds, selenophenes, according to preliminary studies carried out in this laboratory, give Stern–Volmer plots in the shape of segments of a parabola (**14c**) suggesting the formation of an exciplex between benzophenone and the heterocycle. The mathematical equation for these curves should be quadratic in [Q], as has been verified by testing the data with a quadratic polynomial regression program [16]. This situation is commonly encountered when the quencher employed quenches two excited states [17]. However, for $k_q = 10^{10}$ mol^{-1} s^{-1}, which is characteristic of diffusion-controlled quenching, it is accepted [18] that a quencher concentration [Q] of 1 M is required to quench a species with $\tau = 10^{-10}$ s. For S_1 of benzophenone at room temperature, since curves similar to those in fig. 3 of Ref. [12] are obtained for $[Q] = 10^{-2}$ M, it is very unlikely that selenophene is an efficient quencher of the S_1 state of benzophenone. It would be more reasonable to assume that the only excited species in addition to the triplet state of benzophenone that can be quenched under these conditions is an exciplex formed

Table 1
Results obtained from the (2+2) cycloaddition reactions

Heterocycle	Benzophenone alone	Dimethylmaleic anhydride or dimethylmaleimide plus benzophenone
Selenophene[a]	No reaction	1 : 1 and 2 : 1[b] adducts
2-Methylselenophene	Oxetane (**15**)	1 : 1 adduct
3-Methylselenophene	Oxetane (**16**)	Maleimide dimer + polymer
3,4-Dimethylselenophene	No reaction	1 : 1 and 2 : 1[c] adducts
2,5-Dimethylselenophene	Oxetane	Oxetane
cis-Selenophthene		1 : 1 adduct
trans-Selenophthene		1 : 1 and 2 : 1 adducts
Tellurophene	No reaction	
Benzo(b)tellurophene	Oxetane	Oxetane

[a] In the presence of citraconic anhydride yields 1 : 1 adducts.
[b] Three geometrical isomers.
[c] One geometrical isomer.

between triplet excited benzophenone and a ground state molecule of the corresponding selenophene derivative.

As shown in Table 1, selenophene yields a 2+2 cycloaddition product, but is inert to oxetane formation. 2-Methylselenophene and 3-methylselenophene yield either the dimer of the maleimide or a single 1 : 1 adduct, but in the absence of maleimide they yield an oxetane. Finally, 2,5-dimethylselenophene only yields oxetanes either in the presence or absence of maleimide. These observations can be explained as follows.

1. The formation of either the oxetane or the 2+2 adduct depends on kinetic factors and oxetane formation competes well with inefficient energy transfer.
2. The exciplexes between the electron-rich heteroarene and the electron-deficient benzophenone are more or less tightly held according to the ionization potential of the heterocycle and this factor influences the course of the reaction.

The mechanistic aspects of this reaction will be carefully studied and will be the subject of a future publication.

cis-Selenophthene yields only the 1 : 1 adduct (**6**) with both dimethylmaleic anhydride and maleimide, and *trans*-selenophthene affords both the 1 : 1 (**7**) and 2 : 1 (**8**) adducts. The ^{13}C NMR spectrum of the latter reveals the presence of more than three, perhaps six, possible isomers. Its poor solubility in common NMR solvents and in dimethylsulphoxide means that their presence can only be inferred. No attempts have been made to draw any structures. Perhaps pertinent substitutions on the central two ring nucleus may reduce the number of possible isomers as observed for 3,4-dimethylselenophene. Then it may be possible to speculate or make accurate assignments of the stereochemistry.

Finally, tellurophene has been used in two types of cycloaddition reaction in this laboratory. First, it was tested unsuccessfully as a substrate for excited benzophenone to form an oxetane. Presumably the large tellurium atom is extruded from the ring; the heterocycle is not recovered as happens in other instances when the heterocycle under study is inert to the reaction. However, tellurophene has been used in the less common (3+4) cycloaddition reaction as a substrate for dibromoketones, which thermally and photochemically in the presence of iron nonacarbonyl yields (2,3,0)bicyclic structures containing a seven-membered ring with the heteroatom at the bridgehead [19]. Since these compounds are very stable, it was proposed to use tellurophene as a benzo(b)-fused heterocycle in an attempt to take advantage of the additional stability expected from the aromatic ring fused to the heterocyclic nucleus. Thus when benzo(b)tellurophene was irradiated in the presence of benzophenone and 2,3-dimethylmaleimide, oxetane **19** was the only product of the reaction. When benzo(b)tellurophene was irradiated in the presence of benzophenone alone, oxetane **19** was still the only product isolated.

The NMR spectrum of oxetane **19** exhibits, in addition to the aromatic resonances (multiplet between 7.30 and 7.50 ppm), an AB system where the doublet at 8.7 ppm corresponds to the proton at C-1 which, because of its position between the Te and O atoms, resonates at a very low field; another doublet appears at 7.9 ppm and may be assigned to the proton at C-5. In the mass spectrum, the tellurium isotopic distribution was observed whenever the mass fragment contained the tellurium atom.

The mass spectrum revealed that all of the cycloaddition products were first degraded via a retro ($2\pi+2\pi$) process, as reported previously for analogous compounds, to the initial components (heterocycle and maleic anhydride derivative), each of which subsequently underwent its own characteristic fragmentation [6,20]. Consequently, the mass spectrum of the adducts virtually consists of the sum of the two spectra corresponding to the starting materials, e.g. scheme 2 in Ref. [6].

Table 1 summarizes the results obtained from the (2+2) cycloaddition reactions.

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