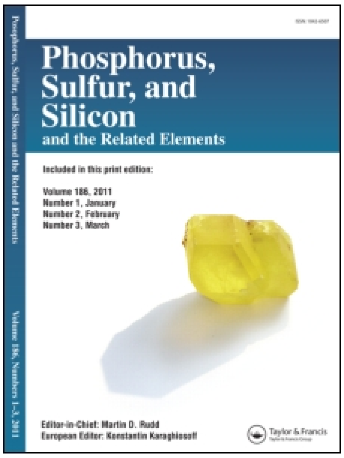

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# SYNTHESIS OF NEW BIS(2,3-DISUBSTITUTED CYCLOPROPYL)BENZENES AND PYRIDINES

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# SYNTHESIS OF NEW BIS(2,3-DISUBSTITUTED CYCLOPROPYL)BENZENES AND PYRIDINES

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Some novel bis(2,3-disubstituted cyclopropyl)benzenes and pyridines have been synthesized by the cycloaddition of arylthiocarbenes to some Michael acceptors under phase transfer conditions. The structures of the new compounds were established by IR, $^1H$ NMR, $^{13}C$ NMR and mass spectral data.

*Key words:* Arylthiocarbenes; bis(cyclopropyl)benzenes and pyridines; cycloaddition; Michael acceptors; phase transfer catalysis.

## INTRODUCTION

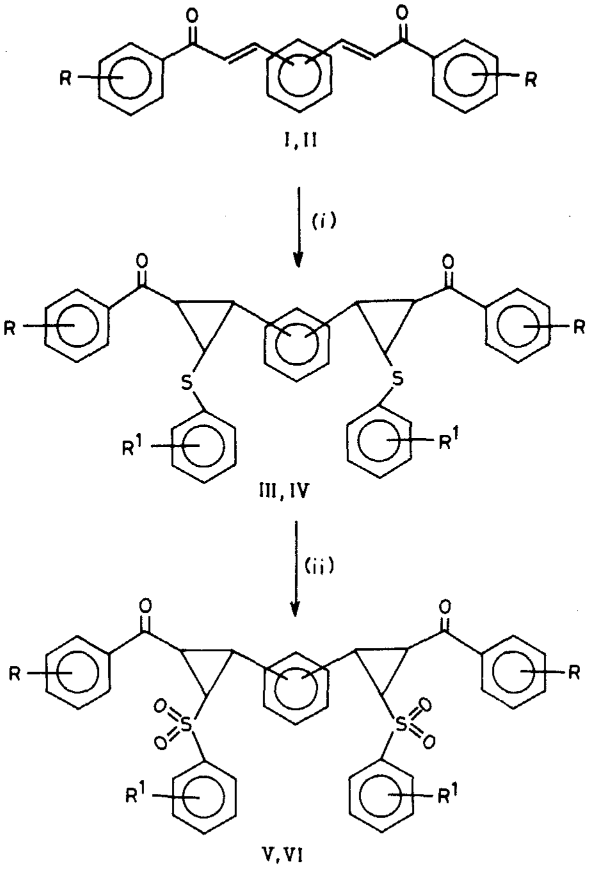
The cyclopropyl moiety is a basic structural unit in a wide range of naturally occurring compounds in plants and in micro-organisms. A number of biologically important compounds contain cyclopropyl group.[1] The most significant property of a cyclopropanoid is manifested in chrysanthemate moiety which is a constituent unit in a number of pyrethroid insecticides.[2] Moreover, several cyclopropanoids were known to possess pharmacological properties.[3] Apart from this, compounds having a sulfonyl moiety were also known to be bio-active.[4] Hence, in recent years much attention is being paid to the synthesis of newer cyclopropanoids containing a sulfonyl functionality, by a viable and facile method.

Among the important methods[5] known for the synthesis of cyclopropyl sulfones, the stereospecific addition of phenylthiocarbene to carbon—carbon double bonds provides a general synthetic route.[5,6] Several workers have reported cyclopropanation of different unsaturated systems with arylthiocarbenes in homogeneous solutions.[7] This method, however, requires rigorous reaction conditions leading to the formation of products in poor yields.

The phase transfer catalysis method which is of recent origin has been very much exploited for the cyclopropanation of olefins with carbenes.[8] However, reports about the utility of arylthiocarbenes for a similar purpose under two-phase reaction system are scanty.[5,9–11] Our continued interest in the synthesis of potential bioactive cyclopropanes led us to plan for the synthesis of novel bis(2,3-disubstituted cyclopropyl)benzenes and pyridines under the above mentioned reaction conditions.

## RESULTS AND DISCUSSION

The substrates 3,3′-(1,3- and 1,4-phenylene)bis(1-aryl-2-propen-1-ones) (**I** and **II**), 1,1′-(1,3- and 1,4-phenylene) and (2,6-pyridylene)bis(3-aryl-2-propen-1-ones) (**VII**, **VIII** and **IX**) have been treated with aryl chloromethyl sulfides in the presence of benzyltriethylammonium chloride (BTEAC) in 50% aqueous sodium hydroxide and methylene chloride at laboratory temperature to obtain 1,1′-(1,3- and 1,4-phenylene)bis[2-aroyl-3-(arylthio)cyclopropanes] (**III** and **IV**) and 1,1′-isophthaloyl/terephthaloyl/(2,6-pyridinediyl)bis[2-aryl-3-(arylthio)cyclopropanes] (**X**, **XI** and **XII**), respectively (Schemes 1 & 2 and Table I). Subsequent oxidation of the latter with 30% hydrogen peroxide in glacial acetic acid yielded the corresponding sulfonylcyclopropanes, **V**, **VI**, **XIII**, **XIV** and **XV** (Table II).



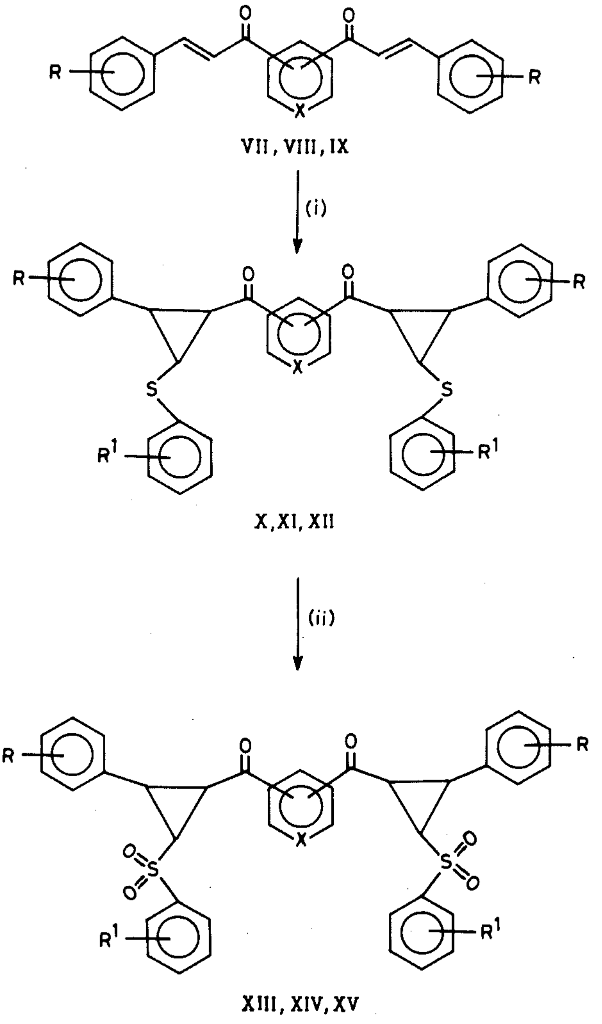
(i) $R^1C_6H_4SCH_2Cl$ / $CH_2Cl_2$ / 50% aq·NaOH / BTEAC

(ii) $H_2O_2$ / AcOH

Where in I $R{-}C_6H_4COCH{=}CH{-}$ at 1-and 3-positions

II $R{-}C_6H_4COCH{=}CH{-}$ at 1-and 4-positions

SCHEME 1



(i) $R^1C_6H_4SCH_2Cl / CH_2Cl_2/50\%$ aq·NaOH/BTEAC

(ii) $H_2O_2$ / AcOH

Where in X $X=CH$, $R-C_6H_4CH=CH-CO-$ at 1-and 3-positions

XI $X=CH$, $R-C_6H_4CH=CH-CO-$ at 1-and 4-positions

XII $X=N$, $R-C_6H_4CH=CH-CO-$ at 2-and 6-positions

SCHEME 2

The arylthiocarbenes are obviously generated in situ under phase transfer conditions more effectively from aryl chloromethyl sulfides. In this method fairly high yields were obtained under simpler experimental conditions involving shorter reaction times when compared with that of Schoellkopf *et al.*[6] The method of preparation of substrates indicate that they possess trans-geometry.[12] This geometry is considered to have been retained in the formation of new bis(cyclopropanes) in our present investigation.[16,19–21]

TABLE I

Melting points and analytical data of **III**, **IV**, **X**, **XI** and **XI**

| No. | | R | R$^1$ | Yield (%) | m.p. (°C) | Calcd.% C | H | Found % C | H |
|---|---|---|---|---|---|---|---|---|---|
| III | a | H | H | 82 | 88-89 | 78.32 | 5.19 | 78.48 | 5.28 |
| | b | H | 4-Cl | 88 | 106-107 | 70.04 | 4.33 | 70.22 | 4.48 |
| | c | 4-OCH$_3$ | 3,4-Cl$_2$ | 76 | 122-123 | 61.55 | 3.87 | 61.40 | 3.94 |
| | d | 4-OC$_2$H$_5$ | 4-CH$_3$ | 69 | 128-129 | 75.61 | 6.06 | 75.82 | 6.16 |
| | e | 4-Cl | H | 81 | 146-147 | 70.04 | 4.33 | 69.87 | 4.21 |
| IV | a | H | H | 89 | 118-119 | 78.32 | 5.19 | 78.54 | 5.26 |
| | b | H | 4-Cl | 84 | 122-123 | 70.04 | 4.33 | 70.28 | 4.46 |
| | c | 4-OCH$_3$ | 3,4-Cl$_2$ | 76 | 177-178 | 61.55 | 3.87 | 61.72 | 4.04 |
| | d | 4-OC$_2$H$_5$ | 4-CH$_3$ | 78 | 167-168 | 75.61 | 6.06 | 75.33 | 6.14 |
| | e | 4-Cl | H | 81 | 181-182 | 70.04 | 4.33 | 70.31 | 4.56 |
| X | a | H | H | 73 | 80-81 | 78.32 | 5.19 | 78.51 | 5.26 |
| | b | 4-CH$_3$ | 3,4-Cl$_2$ | 70 | 93-94 | 58.62 | 3.69 | 58.80 | 3.62 |
| | c | 4-OCH$_3$ | H | 74 | 123-124 | 74.74 | 5.33 | 74.56 | 5.46 |
| | d | 4-Cl | 4-CH$_3$ | 82 | 142-143 | 70.68 | 4.75 | 70.90 | 4.84 |
| | e | 2,4-(OCH$_3$)$_2$ | 4-Cl | 80 | 98-99 | 65.36 | 4.70 | 65.55 | 4.61 |
| XI | a | H | H | 78 | 125-126 | 78.32 | 5.19 | 78.54 | 5.25 |
| | b | 4-CH$_3$ | H | 74 | 141-142 | 78.65 | 5.61 | 78.88 | 5.70 |
| | c | 4-OCH$_3$ | 4-Cl | 71 | 130-131 | 67.50 | 4.53 | 67.71 | 4.62 |
| | d | 4-Cl | 3,4-Cl$_2$ | 83 | 147-148 | 57.81 | 3.06 | 58.04 | 3.17 |
| | e | 2,4-Cl$_2$ | 4-CH$_3$ | 75 | 127-128 | 58.62 | 3.69 | 58.72 | 3.76 |
| XII | a | H | H | 81 | 141.142 | 76.13 | 5.01 | 76.29 | 4.95 |
| | b | 4-CH$_3$ | H | 76 | 154-155 | 76.56 | 5.44 | 76.75 | 5.51 |
| | c | 4-OCH$_3$ | 3,4-Cl$_2$ | 71 | 140-141 | 59.93 | 3.74 | 60.17 | 3.85 |
| | d | 4-Cl | 4-CH$_3$ | 69 | 157-158 | 68.81 | 4.59 | 68.98 | 4.52 |
| | e | 2,4-(OCH$_3$)$_2$ | 4-Cl | 73 | 161-162 | 63.73 | 4.57 | 63.88 | 4.64 |

TABLE II

Melting points and analytical data of **V**, **VI**, **XIII**, **XIV** and **XV**

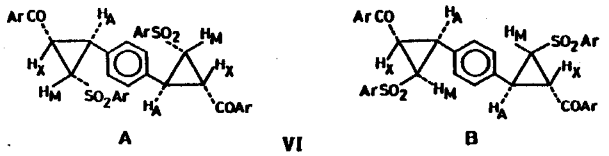
| No. | | R | R$^1$ | Yield (%) | m.p. (°C) | Calcd.% C | H | Found % C | H |
|---|---|---|---|---|---|---|---|---|---|
| V | a | H | H | 78 | 143-144 | 70.57 | 4.68 | 70.80 | 4.75 |
| | b | H | 4-Cl | 82 | 228-229 | 63.78 | 3.94 | 63.98 | 4.09 |
| | c | 4-OCH$_3$ | 3,4-Cl$_2$ | 73 | 202-203 | 56.88 | 3.58 | 57.04 | 3.70 |
| | d | 4-OC$_2$H$_5$ | 4-CH$_3$ | 67 | 196-197 | 69.27 | 5.55 | 69.43 | 5.63 |
| | e | 4-Cl | H | 87 | 240-242 | 63.78 | 3.94 | 63.98 | 4.09 |

TABLE II (*Continued*)

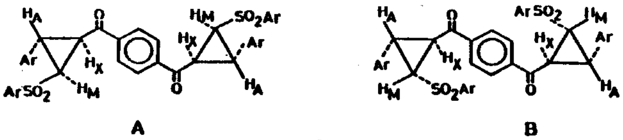
| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| VI | a | H | H | 78 | 204-205 | 70.57 | 4.68 | 70.44 | 4.80 |
| | b | H | 4-Cl | 81 | 210-211 | 63.78 | 3.94 | 63.96 | 4.11 |
| | c | 4-$OCH_3$ | 3,4-$Cl_2$ | 73 | 246-247 | 56.88 | 3.58 | 57.11 | 3.70 |
| | d | 4-$OC_2H_5$ | 4-$CH_3$ | 70 | 231-232 | 69.27 | 5.55 | 69.46 | 5.46 |
| | e | 4-Cl | H | 84 | 253-254 | 63.77 | 3.94 | 63.94 | 4.16 |
| XIII | a | H | H | 73 | 142-143 | 70.57 | 4.68 | 70.77 | 4.61 |
| | b | 4-$CH_3$ | 3,4-$Cl_2$ | 64 | 181-182 | 54.38 | 3.42 | 54.52 | 3.65 |
| | c | 4-$OCH_3$ | H | 68 | 198-199 | 67.97 | 4.85 | 68.14 | 4.92 |
| | d | 4-Cl | 4-$CH_3$ | 78 | 246-248 | 64.60 | 4.34 | 64.78 | 4.41 |
| | e | 2,4-$(OCH_3)_2$ | 4-Cl | 72 | 174-175 | 60.36 | 4.34 | 60.17 | 4.47 |
| XIV | a | H | H | 72 | 229-230 | 70.57 | 4.68 | 70.42 | 4.60 |
| | b | 4-$CH_3$ | H | 67 | 243-244 | 71.19 | 5.08 | 71.36 | 5.14 |
| | c | 4-$OCH_3$ | 4-Cl | 70 | 236-237 | 61.93 | 4.16 | 61.72 | 4.24 |
| | d | 4-Cl | 3,4-$Cl_2$ | 82 | 248-249 | 53.48 | 2.83 | 53.63 | 2.89 |
| | e | 2,4-$Cl_2$ | 4-$CH_3$ | 78 | 181-182 | 54.38 | 3.42 | 54.53 | 3.51 |
| XV | a | H | H | 73 | 202-203 | 68.61 | 4.51 | 68.73 | 4.56 |
| | b | 4-$CH_3$ | H | 78 | 217-218 | 69.31 | 4.92 | 69.52 | 5.01 |
| | c | 4-$OCH_3$ | 3,4-$Cl_2$ | 84 | 236-238 | 55.40 | 3.46 | 55.26 | 3.54 |
| | d | 4-Cl | 4-$CH_3$ | 80 | 224-225 | 62.90 | 4.20 | 63.14 | 4.28 |
| | e | 2,4-$(OCH_3)_2$ | 4-Cl | 71 | 240-242 | 58.85 | 4.22 | 59.02 | 4.29 |

The IR spectra of the compounds III–V and X–XV exhibited a band in the region 1095–1070 (νS-aryl),[13] 1030–1010 (ν ring deformation)[5,10,11] and 1680–1660 (ν C═O)[10,14,15] cm$^{-1}$. The compounds V, VI, XIII, XIV and XV displayed very strong bands in the regions 1335–1315 and 1155–1125 cm$^{-1}$ characteristic of sulfonyl group.[5,10,16]

The structures of VI and XIV are taken as representative examples of each series to correlate the NMR data. It is obvious that various confirmational isomers are possible for VI and XIV by the rotation of one of the cyclopropanes with respect to the other. The different conformers so visualized possess one or the other possible elements of symmetry rendering them as optically inactive. The most reasonable structures amongst them can be assumed as shown below:



VI



XIV

TABLE III

PMR spectral data of **V**, **VI**, **XIII**, **XIV** and **XV**

| S.No. | $^1H$ NMR ($CDCl_3$) δ, ppm | | | Coupling constants, Hz | | |
|---|---|---|---|---|---|---|
| | $H_A$ | $H_M$ | $H_X$ | $J_{AM}$ | $J_{AX}$ | $J_{MX}$ |
| V a | 4.34 | 4.01 | 3.55 | 10.01 | 5.92 | 5.54 |
| b | 4.44 | 4.08 | 3.64 | 10.00 | 5.94 | 5.55 |
| d | 4.25 | 3.89 | 3.46 | 10.03 | 5.93 | 5.55 |
| VI a | 4.40 | 4.10 | 3.71 | 10.04 | 5.97 | 5.62 |
| c | 4.51 | 4.18 | 3.74 | 10.06 | 5.98 | 5.62 |
| e | 4.29 | 3.96 | 3.60 | 10.05 | 5.97 | 5.63 |
| XIIIa | 4.48 | 4.19 | 3.76 | 10.01 | 5.94 | 5.62 |
| b | 4.55 | 4.26 | 3.85 | 10.04 | 5.96 | 5.63 |
| c | 4.37 | 4.08 | 3.66 | 10.02 | 5.95 | 5.64 |
| XIVa | 4.44 | 4.16 | 3.74 | 10.07 | 6.00 | 5.66 |
| b | 4.31 | 3.99 | 3.58 | 10.05 | 5.98 | 5.65 |
| e | 4.46 | 4.20 | 3.77 | 10.06 | 6.01 | 5.67 |
| XV a | 4.56 | 3.73 | 3.54 | 10.12 | 6.03 | 5.69 |
| b | 4.48 | 3.67 | 3.46 | 10.13 | 6.02 | 5.68 |

The three methine protons of the cyclopropane ring are non-equivalent and hence they should display an AMX pattern. In fact the $^1H$ NMR spectra exhibited the same and the integration data indicated that the two cyclopropane rings are identical. Each methine proton appeared as a doublet of doublet at 4.25–4.56, 3.67–4.26 and 3.46–3.85 ppm (Table III). The signal at higher $\delta_H$ value is assigned to $H_A$ which is more deshielded than $H_M$ or $H_X$.[10,17] Among the latter, the $H_M$ appeared downfield in comparison to $H_X$ as a result of the more deshielding effect of the sulfonyl group compared to the carbonyl group.[10,18] The coupling constants measured for these compounds are in the range: $J_{AX}$ = 5.92–6.03, $J_{AM}$ = 10.00–10.07, $J_{MX}$ = 5.54–5.69 Hz. Hence, it may be derived from the J values that $H_A$

TABLE IV

CMR spectral data of **V**, **VI**, **XIII**, **XIV** and **XV**

| S.No. | Chemical shifts measured to downfield from tetramethylsilane in δ, ppm | | | |
|---|---|---|---|---|
| | C-1 | C-2 | C-3 | C-4 |
| V a | 29.94 | 37.31 | 39.98 | 193.93 |
| c | 30.27 | 37.74 | 39.61 | 194.68 |
| e | 30.16 | 37.52 | 39.43 | 194.26 |
| VI a | 30.02 | 37.62 | 39.46 | 194.22 |
| b | 30.13 | 37.69 | 39.51 | 194.37 |
| d | 29.83 | 37.18 | 38.97 | 193.84 |
| XIIIa | 37.74 | 30.16 | 39.53 | 194.47 |
| b | 38.08 | 30.46 | 39.87 | 194.95 |
| c | 37.47 | 29.93 | 39.21 | 194.04 |
| XIVa | 37.53 | 29.89 | 39.31 | 194.08 |
| b | 37.07 | 29.62 | 38.89 | 193.69 |
| c | 37.81 | 29.75 | 39.04 | 193.88 |
| XV a | 37.96 | 30.54 | 39.75 | 194.59 |
| b | 37.53 | 30.38 | 39.38 | 194.31 |
| c | 38.13 | 30.81 | 39.94 | 194.98 |
| d | 38.05 | 30.69 | 39.89 | 194.79 |

and $H_M$ are cis while $H_X$ is trans oriented with respect to $H_A$ and $H_M$ in all these compounds. This obviously shows that the isomer **B** truly represents **VI** and **XIV**.

The $^{13}C$ NMR spectra showed chemical shift values for carbons, of the cyclopropane ring around 30.0 (C-1), 37.0 (C-2) and 39.0 (C-3) ppm in case of **V** and **VI** while 37.0 (C-1), 30.0 (C-2) and 39.0 (C-3) for **XIII, XIV** and **XV** (Table IV). The $\delta_C$ values exhibited around 193.0 ppm are due to the carbonyl carbons.[19]

## TABLE V

Mass spectral data of **V** and **VI**

| S.No. | $M^{+\cdot}$ | $[M-SO_2]^{+\cdot}$ | $[M-RC_7H_4O]^+$ | $[M-2SO_2]^{+\cdot}$ | $[RR^1C_{22}H_{15}O_3S]^+$ | $[RR^1C_{22}H_{15}O]^+$ | $[RR^1C_{16}H_{11}O_3S]^+$ | $[RC_{10}H_6O]^+$ | $[RC_7H_4O]^+$ | $[RC_6H_4]^+$ |
|---|---|---|---|---|---|---|---|---|---|---|
| **V** a | 646(2) | 582(7) | 541(3) | -- | 361(16) | 297(29) | 285(24) | 143(25) | 105(100) | 77(57) |
| b | -- | 650(5) | 609(8) | 586(7) | 395(18) | 331(22) | 319(28) | 143(26) | 105(100) | 77(64) |
| e | -- | 650(2) | 575(5) | 586(9) | 395(24) | 331(17) | 319(36) | 177(19) | 139(100) | 111(43) |
| **VI** a | 646(6) | 582(5) | 541(3) | 518(8) | 361(23) | 297(31) | 285(21) | 143(29) | 105(100) | 77(52) |
| b | -- | 650(8) | 609(11) | 586(6) | 395(28) | 331(22) | 319(14) | 143(42) | 105(100) | 77(43) |
| d | -- | 698(3) | 613(5) | 634(17) | 419(13) | 355(52) | 343(34) | 187(18) | 149(100) | 121(63) |

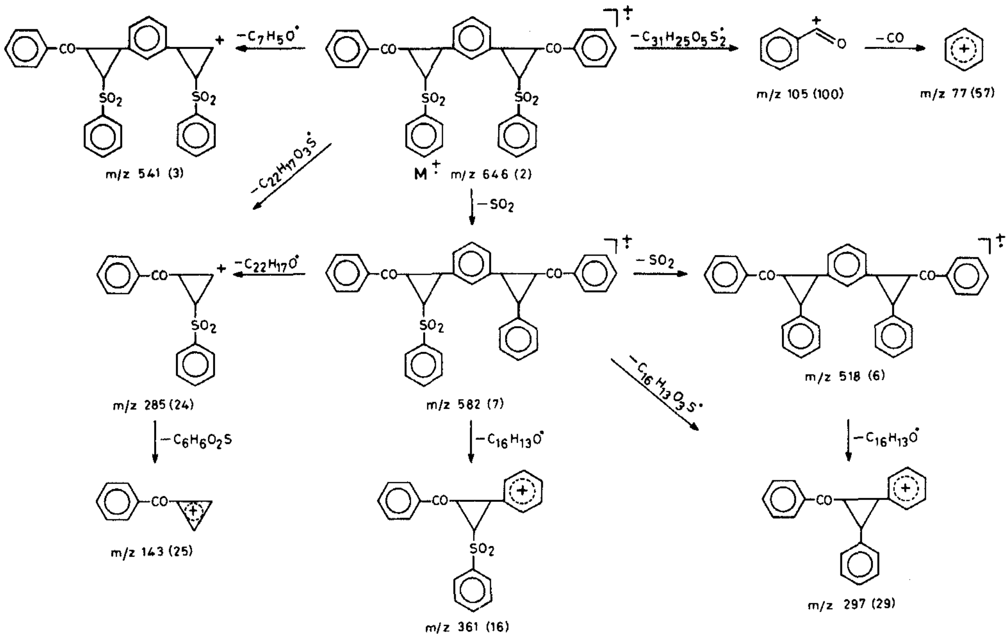
## TABLE VI

Mass spectra data of **XIII**, **XIV** and **XV**

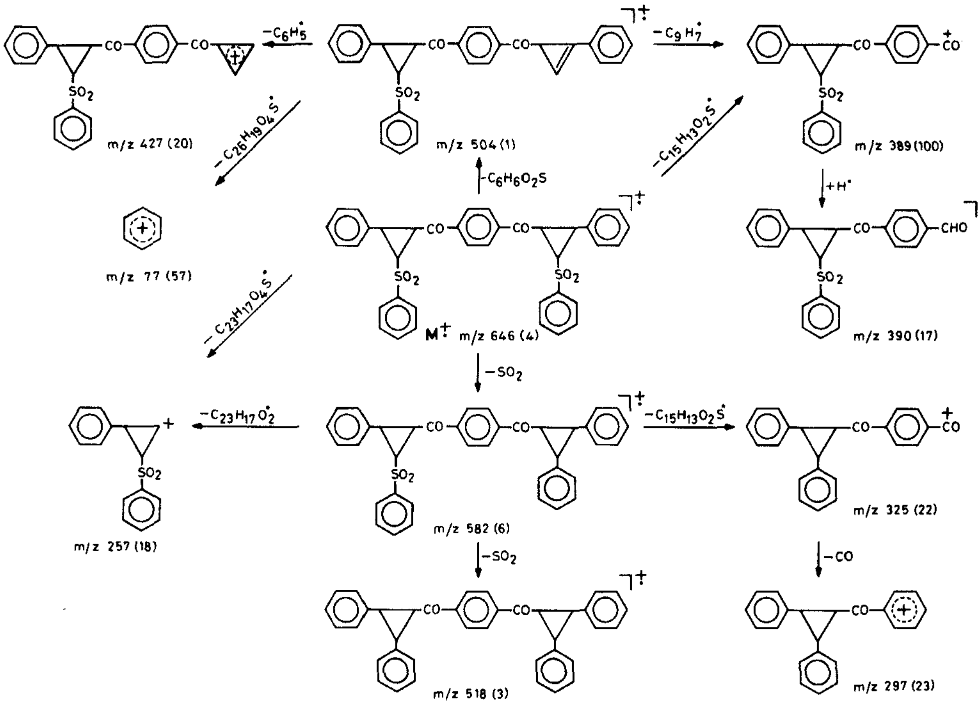
| S.No. | $M^{+\cdot}$ | $[M-SO_2]^{+\cdot}$ | $[M-R^1C_6H_5O_2S]^{+\cdot}$ | $[M-RR^1C_{12}H_9O_2S]^+$ | $[M-RR^1C_{15}H_{11}O_2S]^+$ | $[RR^1C_{28}H_{15}O_2]^+$ | $[RR^1C_{15}H_{11}O_2S]^+$ | $[RC_6H_4]^+$ | $[R^1C_6H_4]^+$ |
|---|---|---|---|---|---|---|---|---|---|
| **XIIIa** | 646(3) | 582(4) | -- | 427(15) | 389(100) | 325(29) | 257(23) | 77(54) | 77(54) |
| d | -- | 678(4) | 586(3) | 475(18) | 437(100) | 373(34) | 305(28) | 111(33) | 91(52) |
| **XIV** a | 646(4) | 582(6) | 504(1) | 427(20) | 389(100) | 325(22) | 257(18) | 77(57) | 77(57) |
| b | 674(2) | 610(5) | 532(4) | 441(17) | 403(100) | 339(18) | 271(23) | 91(55) | 77(43) |
| c | -- | -- | 598(2) | 491(24) | 453(100) | 389(16) | 321(26) | 107(58) | 111(28) |
| **XV** a | 647(1) | 583(8) | 505(2) | 428(13) | 390(100) | 326(18) | 257(21) | 77(48) | 77(48) |
| b | -- | 611(2) | 533(3) | 442(14) | 404(100) | 340(24) | 271(32) | 91(62) | 77(58) |
| d | -- | 679(5) | 587(2) | 476(9) | 438(100) | 374(28) | 305(32) | 111(36) | 91(63) |

Note: Values in the parenthesis indicate the percentage intensity.

-- Indicates the absence of a peak.



SCHEME 3



SCHEME 4

The 70 eV mass spectra of these compounds showed a low intense $M^{+\cdot}$ ion peaks which are in conformity with their chemical composition (Tables V and VI). Some of them did not show molecular ion peaks. However, M-$SO_2$ or M-$2SO_2$ peak is observed in such instances. A facile elimination of one or two molecules of $SO_2$ from the $M^{+\cdot}$ ion is observed in all these cases.[20] An aroyl cation appeared as a base peak[21] in **V** and **VI** whereas, 4-[[2-aryl-3-(arylsulfonyl)cyclopropyl]carbonyl]benzoyl cation appeared in **XIII, XIV** and **XV**. The **XV** exhibited $M^{+\cdot}$ ion with an odd mass value indicating the presence of nitrogen.[22] The other fragmented ions formed are shown in Schemes 3 and 4 which confirms their structures.

## EXPERIMENTAL

All the melting points were determined on a Mel-Temp apparatus and are uncorrected. The IR spectra were recorded on a Perkin-Elmer Grating infrared spectrophotometer model 337. The $^1H$ and $^{13}C$ NMR spectra were obtained in $CDCl_3$ on GE NMR Omega and Bruker 500 MHz spectrometers with tetramethylsilane as an internal standard. The mass spectra were recorded on a Finnigan Mat 1210 instrument at 70 eV. Microanalyses were performed by Central Drug Research Institute, Lucknow, India.

*1,1'-(1,3-/1,4-Phenylene)bis[2-aroyl-3-(arylsulfonyl)cyclopropanes]* (**III** *and* **IV**), *1,1'-isophthaloyl-/terephthaloyl-/(2,6-pyridinediyl)bis[2-aryl-3-(arylsulfonyl)cyclopropanes]* (**X, XI** *and* **XII**)*: General procedure:* A mixture of 5 mmol of **I/II/VII/VIII/IX**,[12] 11 mmol of aryl chloromethyl sulfide and 40 ml of methylene chloride was stirred with 30 ml of 50% aqueous sodium hydroxide to obtain a clear two-phase system. Then, 100 mg (0.4 mmol) of benzyltriethylammonium chloride was added and stirring was continued at room temperature for 14–16 hr. After completion of the reaction, the contents were diluted with water; the organic layer was separated, washed with water, brine and then dried. Removal of the solvent gave a syrupy substance which was solidified on treatment with 2-propanol. Recrystallization from alcohol or benzene gave pure **III, IV, X, XI** and **XII**. A solution of 10 mmol of the latter in 25 ml of glacial acetic acid was refluxed with 15 ml of 30% hydrogen peroxide for a period of 8–10 hr. The contents were allowed to cool and then poured onto crushed ice with stirring. The solid product separated was collected washed with water dried and recrystallized from 2-propanol or benzene to obtain **V, VI, XIII, XIV** and **XV**, respectively. The purity was ascertained by thin layer chromatography.

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