THE REACTION OF UNSATURATED CARBONYL COMPOUNDS WITH "ACTIVATED" SULFUR (II). FORMATION OF CYCLIC DISULFIDE AND POLYSULFIDES[1]

Ugo Chiacchio, Antonino Corsaro, Venerando Pistarà, Giovanni Purrello,* and Antonio Rescifina

Dipartimento di Scienze Chimiche, Università di Catania, Viale A. Doria 6, 95125 Catania, Italy

Abstract - From the reaction of cinnamylideneacetophenone (**1**), sulfur and triethylamine in suitable solvents at room temperature, phenyl{3-[(*E*)-1-((5-benzoyl-3*H*-1,2-dithiol-3-ylidene)phenylmethyldisulfanyl)-1-phenylmethylidene]-3*H*-1,2-dithiol-5-yl}methanone (**7**) was isolated together with two polysulfurated compounds to which structures of phenyl(7-phenyl-1,2,3-trithiepin-4-yl)methanone (**12**) and phenyl-(8-phenyl-1,2,3,4-tetrathiocin-5-yl)methanone (**13**) were assigned. The obtaining of these new products allows to complete the sequence of the whole process.

Previously we reported that compounds (**2-6**) were isolated from a thiation process of cinnamylideneacetophenone (**1**) with sulfur in the presence of triethylamine[2] (TEA) and a suitable solvent at room temperature[3] (Figure 1).

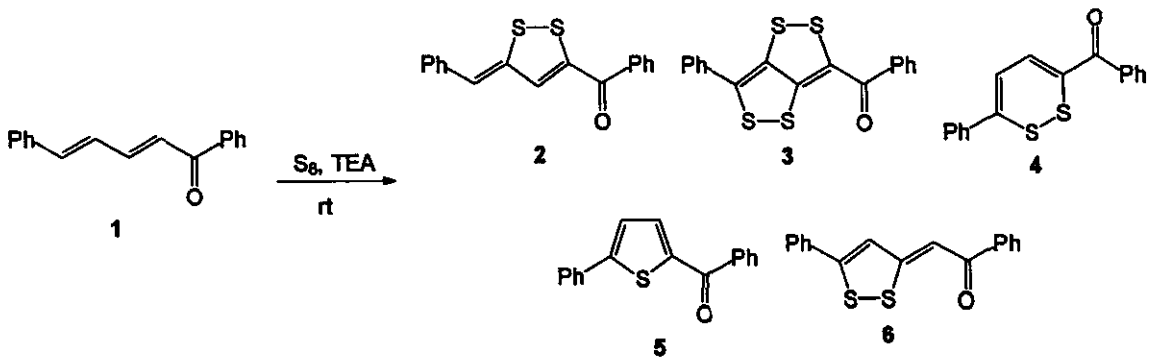


Figure 1

Pursuing in the study of this thiation process we have now been able to isolate some other compounds to which the structures of phenyl{3-[(*E*)-1-((5-benzoyl-3*H*-1,2-dithiol-3-ylidene)phenylmethyldisulfanyl)-1-phenylmethylidene]-3*H*-1,2-dithiol-5-yl}methanone (7), phenyl(7-phenyl-1,2,3-trithiepin-4-yl)methanone (12) and phenyl(8-phenyl-1,2,3,4-tetrathiocin-5-yl)methanone (13) are assigned (Figure 2). All these compounds were formed in the reaction of 1 with sulfur and TEA for well definite times and solvents.

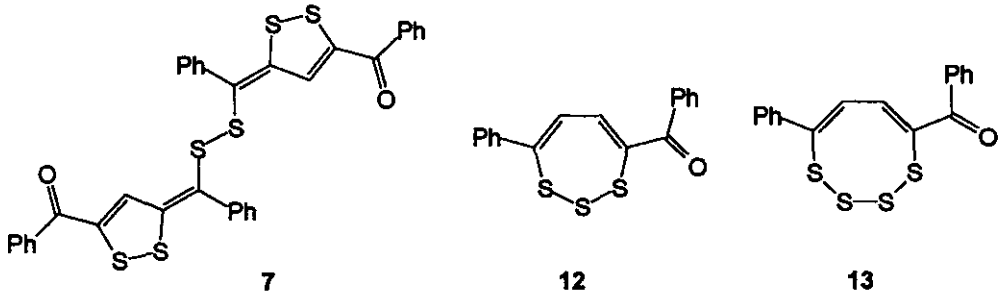


Figure 2

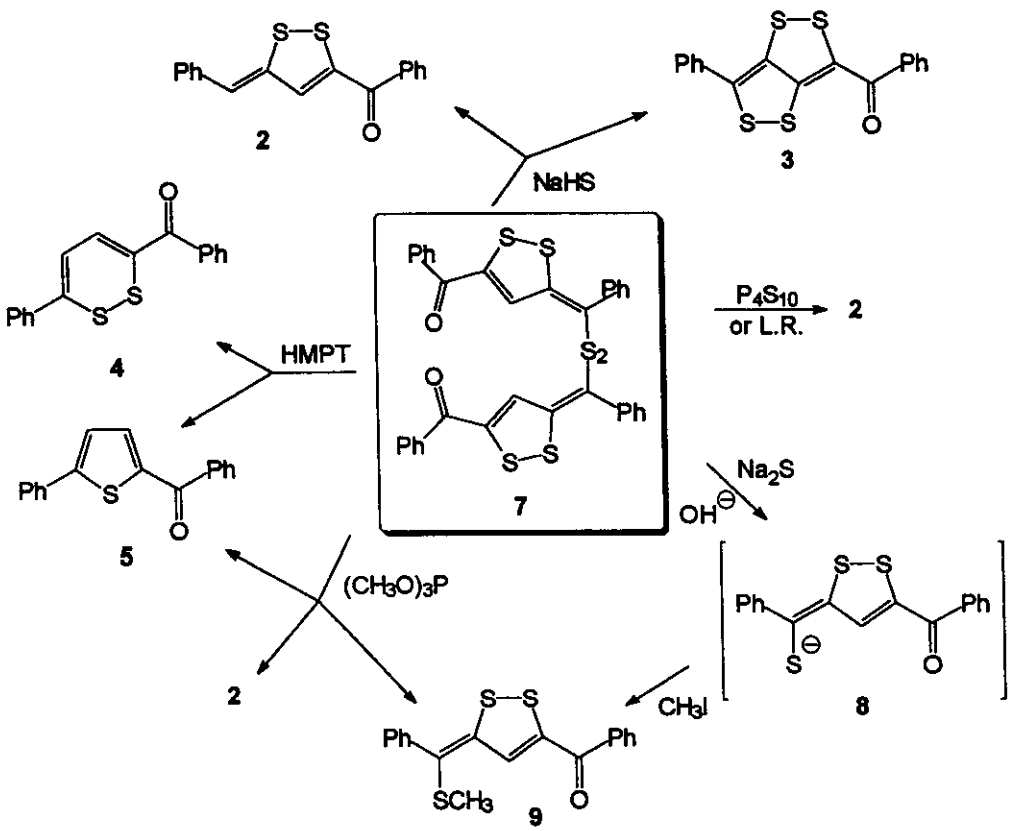
The first of these was isolated as a violet powder, mp 52-53 °C, with a 20 % yield by the chromatography of a reaction mixture obtained in dimethyl sulfoxide for 3 h and it was identified on the basis of its spectral data and chemical behavior. Particularly, the MS spectrum, performed under the FAB technique, exhibits the molecular ion at m/z 655 $(MH)^+$ and a series of fragmentation ions among which the most significant are those deriving from the loss of one and two atoms of sulfur from the molecular ion at m/z 623 $(MH - S)^+$ and m/z 591 $(MH - 2S)^+$, respectively, and the enethiol ion deriving from the disulfide bond scission at m/z 328. The ^1H NMR spectrum presents signals only in the aromatic region. The UV spectrum shows two absorption bands at 538 and 272 mμ.

The disulfide (7) appears in different conformational forms. TLC shows the prevalent presence of three conformers of which one is in minimum amount with respect to the other two. The separation of these different conformers on preparative TLC does not allow their stable isolation because each of them, after extraction, gives the mixture of conformers in the original composition.

The reaction of the disulfide (7) with sodium hydrosulfide leads to the 1,2-dithiole (2) and 1,2-dithiole[4,3-c]1,2-dithiole (3), while that with the Lawesson's reagent (L. R.) or phosphorus decasulfide in toluene at room temperature leads to the 1,2-dithiole (2). Compounds (2) and (3) seem to derive by a scission of the C-S bond rather than by a scission of the S-S bond of 7 and a subsequent intramolecular thiation process on the β position seems to take place for the production of 3. The treatment with hexamethylphosphorous triamide (HMPT) converted the disulfide (7) into compounds (4) and (5). The reaction of the disulfide (7) with sodium sulfide and then iodomethane produces phenyl{3-[(*E*)-1-methylsulfanyl-1-phenylmethylidene)-3*H*-1,2-dithiol-5-yl]}methanone (9)[5] through the ene thiolate (8) (Scheme 1). The

methylthio ether derivative (**9**) is also obtained by treatment of the disulfide (**7**) with trimethyl phosphite (TMP) at room temperature along with the 1,2-dithiole (**2**) and thiophene (**5**) derivatives.

The MS spectrum of **9**, yellow dense oil, shows the molecular ion at m/z 342 $(M)^+$ and other peaks are at m/z 310 $(M - S)^+$, m/z 295 $(M - SMe)^+$ and m/z 105 $(PhCO)^+$. Its ^1H NMR spectrum is characterized by a singlet at δ 2.39 (3H) relative to the methylthio protons and three multiplets centered at δ 7.53 (8H), 7.98 (1H) and 8.1 (2H). The UV spectrum shows four absorption bands at 448, 430s, 344s and 264 mμ.



Scheme 1

After the disulfide (**7**), among numerous yellow products present in low concentration, it was possible to isolate various amounts of some yellow to orange compounds, i.e. **12** and **13**, which were very difficult to obtain in a good grade of purity because their similarity and the thermolability of **13**.[7]

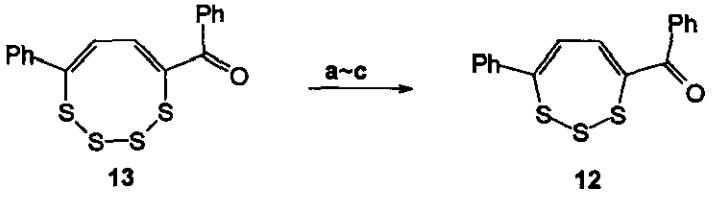
Compounds (**12**) and (**13**) were obtained in higher yields when the reaction mixture of **1** with sulfur and TEA were allowed to react in dimethyl sulfoxide (DMSO) or dimethylformamide (DMF) for 48 or 72 h, respectively.

They were identified from the spectral and chemical data, too. Particularly, the trithiepin (**12**) was isolated

in 4 % yield, as a light yellow needles, mp119-122 °C. Its MS spectrum shows the molecular ions at m/z 328 $(M)^+$ and the following other peaks at m/z 296 $(M - S)^+$, m/z 264 $(M - 2S)^+$, m/z 105 $(PhCO)^+$. The ^1H NMR spectrum shows signals only in the aromatic region at 7.34-7.89 ppm, while the ^{13}C NMR spectrun confirms the presence of three quaternary carbon atoms at 142.27 (C-4), 153.20 (C-7) and 187.85 (C=O) ppm and two olefinic carbon atoms at 123.81 and 135.85 ppm. The UV spectrum shows three absorption bands at 446s, 336 and 260 mμ.[8]

The tetrathiocin (**13**), mp 85 °C, which was obtained in the highest yield (6 %) among polysulfurated compounds, exhibits in the MS spectrum the molecular ion at m/z 360 $(M)^{+\cdot}$ and the following other peaks at m/z 328 $(M - S)^+$, m/z 296 $(M - 2S)^+$, m/z 264 $(M - 3S)^+$. The ^1H NMR spectrum shows signals only in the aromatic region at 7.17-7.83 ppm and the ^{13}C NMR spectrum shows signals corresponding to three quaternary carbon atoms at 141.44 (C-5), 154.43 (C-8), 186.96 (C=O) ppm and two olefinic carbon atoms at 131.82, 140.04 ppm. The UV spectrum shows absorption bands at 448, 428, 332 and 258 mμ.[8]

The tetrathiocin (**13**) in solution slowly loses a sulfur atom at room temperature giving the trithiepin (**12**), which is yielded also by reaction with TMP at room temperature (Scheme 2). This fact that it does not produce any methylthio ether derivative means that a type Arbuzov reaction is not possible for the lack of an acidic hydrogen atom. By heating the tetrathiocin (**13**) in refluxing toluene, the trithiepin (**12**) rapidly can be also obtained.



13 **12**

a = room temperature
b = TMP at room temperature
c = refluxing toluene

Scheme 2

By reaction with dimethyl acetylenedicarboxylate (DMAD) in refluxing benzene the tetrathiocin (**13**) loses two sulfur atoms affording the 1,2-dithiin derivative (**4**) (60%).[9]

Solvent effect

Previously we showed that the product distribution is remarkably affected by the solvent nature and reaction time.[2] In the course of the present study, it has been now verified that no appreciable reaction is observed, even for long periods of time, when cinnamylidenacetophenone (**1**) and sulfur are treated at room temperature with the alone TEA or solvent.[10] If dimethylformamide (DMF) or hexamethylphosphoric

triamide (HMPA) are used as solvents the mixture sulfur-TEA becomes immediately brown, also in the absence of any organic compound. In the presence of **1**, TLC indicates the formation of the dithiole (**2**) after *ca.* 15 min. This compound, however, disappears rapidly (within 2 h in HMPA and 5 h in DMF) turning into the 1,2-dithiole[4,3-*c*]1,2-dithiole (**3**) and disulfide (**7**). By using dimethyl sulfoxide (DMSO) or acetonitrile (MeCN) as solvents the dithiole (**2**) is formed within 2 h; in pyridine (Py) and nitromethane ($MeNO_2$) the dithiole (**2**) is formed within 5 h, in acetone within 7 h and in ethyl acetate within 20 h. In other solvents, such as methanol, ether, tetrahydrofuran and dioxane, the reaction practically does not take place. Also the presence of other reaction products, such as a disulfide (**7**), is more or less transitory.

By examining the reaction in the whole, it clearly appears that the reaction proceeds rapidly when a base, TEA in our case, acts by loosening or breaking the S-S bond of the sulfur molecule and a substantially aprotic solvent, but with high polarity and strong electron-donor ability, is present. This is the case of the highest reaction rate in HMPA, DMF and DMSO (ε = 31.3, 37.3 and 47.0,[11] respectively) which have a stronger availability to act as an electron-donor system in comparison with that in nitromethane and methanol (ε = 36.0 and 33.0,[11] respectively), which are poor electron-donor solvents.

The smallest quickness of reactions in some solvents has different possible causes. On one hand the solvent has a minor ability to act as an electron-donor system - this is the case of acetonitrile (ε = 38.0)[11] and nitromethane (ε = 36.0)[11] - on the other hand, as for example in the case of pyridine (ε = 12.5),[11] the solvent has a low dielectric constant, which is partially compensated by its greatest availability to act as an electron-donor system. This allows to use these solvents, for example pyridine or acetonitrile, for practical purposes where the reaction is required to proceed more slowly for a better separation of someone of reaction products.

However, the process does not complete in all the examined solvents. Thus, for example, the reaction leads to the exclusive formation of small amounts of **2** in ethyl acetate (ε = 6.0)[11] and acetone (ε = 21.0),[11] while the formation of compound (**3**) is not observed in nitromethane. The Figure 3 indicates the persistence times of compound (**2**), (**3**) and (**7**) in different solvents for the reaction of **1** with sulfur and TEA as determined by TLC.

EXPERIMENTAL

All melting points were determined on a Kofler hot-stage apparatus and are uncorrected. Elemental analyses were performed on a Carlo Erba 1106 elemental analyzer. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker WP 80 FT spectrometer using tetramethylsilane as internal standard and deuterochloroform as solvent, unless otherwise stated. IR spectra were recorded on a Perkin Elmer 281 and Synthesis 2000

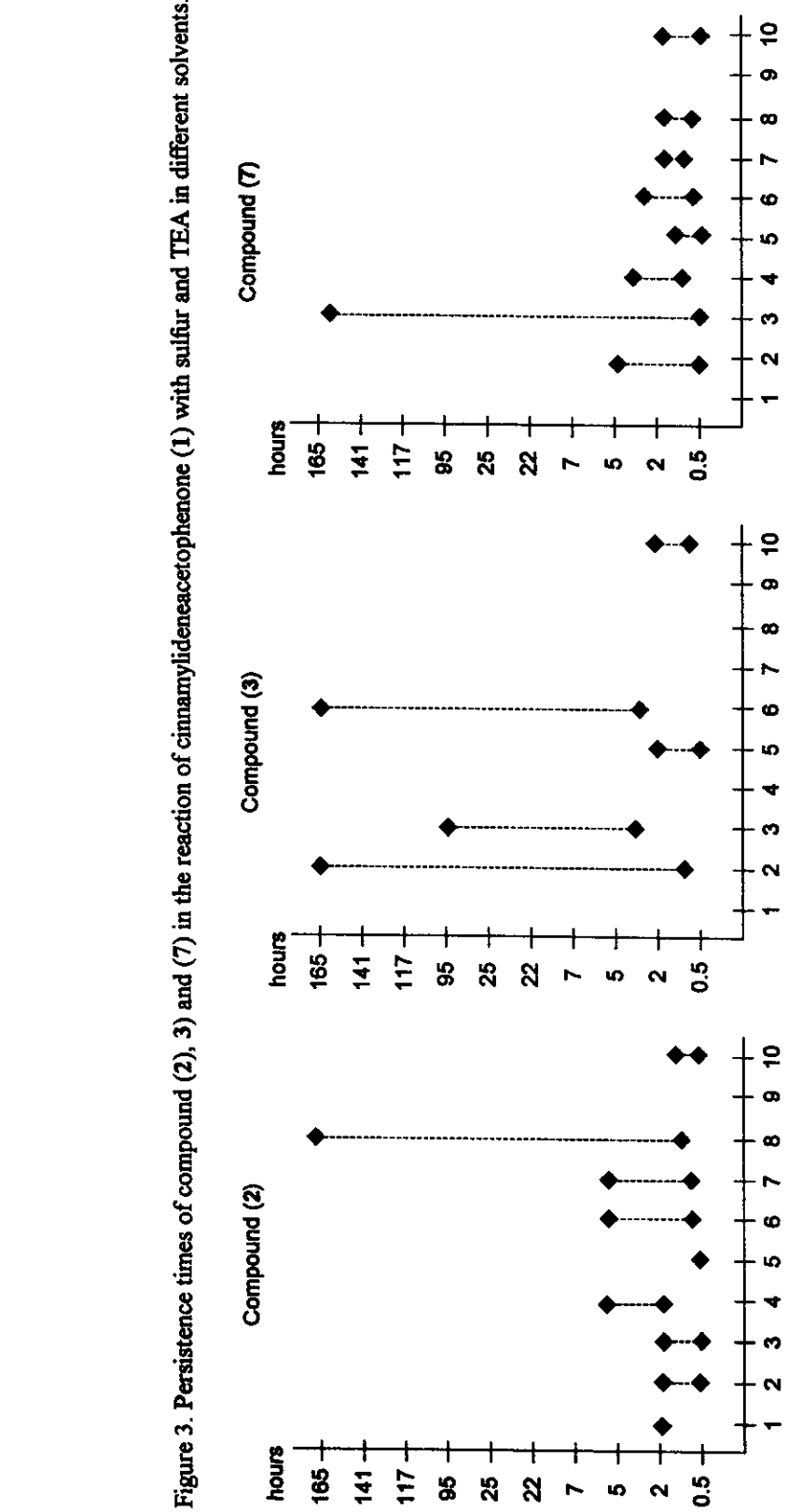


Figure 3. Persistence times of compound (2), 3) and (7) in the reaction of cinnamylideneacetophenone (1) with sulfur and TEA in different solvents.

1 = $MeCO_2Et$; 2 = DMF; 3 = DMSO; 4 = MeCN; 5 = HMPA; 6 = Py; 7 = $MeNO_2$; 8 = MeCOMe; 9 = without solvent; 10 = Morpholine.

spectrophotometer as potassium bromide discs or neat. UV spectra on a HP 8452 A spectrophotometer in dichloromethane solutions. EI and FAB MS spectra were recorded on a VG ZAB-2SE spectrometer. Analytical thin layer chromatographic separation were performed on aluminium plates pre-coated with Merck silica gel 60-F_{254}. Chromatographic separations of reaction mixtures were performed by means of column gravity or flash chromatography using Merck silica gel 60 and in some cases by means of centrifugally enhanced preparative thin layer chromatography (CEPTLC) using plates coated with Merck silica gel 60-PF_{254}. Mixtures of cyclohexane-ethyl acetate were used as eluents.

Starting materials. Cinnamylideneacetophenone (**1**)[12] and compounds (**2-6**)[4] were prepared as previously reported. Solvents have been dried following literature procedures.[13] The identification of samples from different experiments was secured by mixed mps and/or superimposable IR and UV spectra.

Preparation of the disulphide (**7**). Cinnamylidenacetophenone **1** (4.7 g, 20 mmol), sulfur (9.6 g, 0.3 g/at) and triethylamine (10 ml) in dimethyl sulfoxide (20 ml) were allowed to react under stirring at rt. After 3 h the reaction mixture was directly chromatographed by using cyclohexane as initial eluent until all DMSO was eluted. Then, increasing amounts of ethyl acetate were added to cyclohexane and the following fractions were eluted in the order: compounds (**1-5**) in small amounts, which were not furtheron separated, mixtures of yellow compounds containing the trithiepin (**12**), the violet disulfide (**7**) and finally tetrathiocin (**13**). The disulfide (**7**) was purified by repeated flash chromatography and then crystallized from cyclohexane.

In TLC the disulfide (**7**) appears as three distinct purple spots of which one was in minor amount with respect to the other two and which were identified as conformers because the separation on the preparative TLC does not allow a stable isolation of compounds corresponding to each spot. After extraction with dichloromethane of silica gel relative to each spot, TLC of extracts gave a mixture of the same three spots in the original composition.

Phenyl{3-[(E)-1-((5-benzoyl-3H-1,2-dithiol-3-ylidene)phenylmethyldisulfanyl)-1-phenylmethylidene]-3H-1,2-di-thiol-5-yl}methanone (**7**): amorphous purple powder (1.30 g, 20 %), mp 52-53 °C (Anal. Calcd for $C_{34}H_{22}O_2S_6$: C, 62.36; H, 3.39; S, 29.37. Found: C, 62.31; H, 3.32; S, 29.38); λ_{max} (CH_2Cl_2) 538, 272 mμ; ν_{max} (KBr) 1636 cm^{-1}; δ_H ($CDCl_3$) 6.87-7.85 (m, 22 H); MS (FAB): m/z 655 $(MH)^+$, 623 $(MH - S)^+$, 591 $(M - 2S)^+$, 558, 328.

Reaction of the disulfide (7) with sodium sulfide and then with iodomethane. To a refluxing solution of **7**

(0.8 g, 1.2 mmol) in ethanol (50 mL) an aqueous solution (2.5 mL) of sodium sulfide (0.6 g, 7.7 mmol) and sodium hydroxide (0.4 g, 10 mmol) was added. The mixture was refluxed for additional 2 h and then it was cooled in an ice bath. Iodomethane (2 mL, 32 mmol) was added and the mixture was allowed under stirring for 3 h. During this time an orange solid separated which was filtered and the filtrate was evaporated under vacuum. The resulting residue was extracted with benzene (twice, 20 mL) and extracts were dried over sodium sulfate. The filtrate combined with the orange solid was concentrated at reduced pressure and then subjected to flash-chromatography to give *phenyl{3-[(E)-1-methylsulfanyl-1-phenylmethylidene)-3H-1,2-dithiol-5-yl]}methanone* (**9**) as a dense yellow oil (0.42 g, 50 %) (Anal. Calcd for $C_{18}H_{14}OS_3$: C, 63.13; H, 4.12; S, 28.08. Found: C, 63.10; H, 4.13; S 28.11); λ_{max} (CH_2Cl_2) 448, 430s, 344s and 264 mμ; ν_{max} (neat) 1675 cm^{-1}; δ_H ($CDCl_3$) 2.39 (s, 3 H), 7.53 (m, 8 H), 7.98 (m, 1 H) and 8.10 (m, 2 H); MS: m/z 342 $(M)^+$, 310 $(M - S)^+$, 295 $(M - SCH_3)^+$, 105 $(PhCO)^+$.

Reaction of the disulfide (7) with trimethyl phosphite. To a solution of 7 (0.15 g, 0.23 mmol) in benzene (3 mL) trimethyl phosphite (0.15 g, 1.2 mmol) was dropwise added at rt. After 24 h the solvent and the excess of TMP were removed under vacuum and the resulting yellow oil was chromatographed to give, in order of elution, compounds (**2**), (**9**) and (**5**).

Reaction of the disulfide (7) with sodium hydrosulfide. A solution of 7 (0.15 g, 0.23 mmol) and an excess of sodium hydrosulfide in ethanol (20 mL) was refluxed for 20 min. After this time TLC shows the presence of compounds (**2**) and (**3**); the maximum yield of the compound (**2**) was achieved after 40 min. The solvent was removed and the residue was directly chromatographed to give compounds (**2**) and (**3**).

Reaction of the disulfide (7) with HMPT. To a solution of 7 (0.15 g, 0.23 mmol) in anhydrous benzene (1.5 mL) HMPT (0.15 g, 0.92 mmol) was added at rt. After 24 h the solvent and HMPA were removed under vacuum and the resulting solid was subjected to CEPTLC to give compounds (**4**) and (**5**).

Reaction of the disulfide (7) with phosphorous decasulfide or Lawesson's reagent. To a solution of 7 (0.15 g, 0.23 mmol) in toluene (5 mL) an excess of phosphorous decasulfide (0.2 g, 0.45 mmol) or Lawesson's reagent (0.4 g, 0.98 mmol) was added and the mixture was allowed to react at rt under stirring for 12 h. The reaction mixture was then diluted with toluene, washed with water and dried over sodium sulfate. The solvent was removed under vacuum to give a residue which was chromatographed to afford compound (**2**) (0.06 g, 44%).

Preparation of yellow-orange compounds (12) and (13). Cinnamylideneacetophenone (**1**) (9.4 g, 40 mmol), sulfur (19.2 g, 0.6 g/at) and TEA (20 mL) in DMF (30 mL) were allowed to react under stirring at rt for 72 h. If DMSO (30 mL) was used, the reaction time could be reduced to 48 h. Two ways were then followed to separate the different products.

a) The reaction mixture was poured into water under stirring. The separated solid was filtered, washed with water, dissolved in dichloromethane and finally dried over sodium sulfate. After having removed the solvent at reduced pressure, the red residue was repeatedly extracted with pentane, hexane and cyclohexane which removed most of compounds (**4**) and (**5**). The residual mixture was chromatographed to give, in order of elution, the light yellow trithiepin (**12**), which was accompanied by modest amounts of thiophene (**5**) and the yellow-orange tetrathiocin (**13**), impure of the dithiin (**6**), which was the prevailing product of this step of the whole process. These compounds were purified by CEPTCL and in some cases they were further purified by extraction with cyclohexane at rt under prolonged stirring.

b) The reaction mixture was directly subjected to column chromatography by using cyclohexane as initial eluent until all DMF or DMSO was eluted. When increasing amounts of ethyl acetate was added to cyclohexane, the following compounds were isolated in order of elution: compounds (**2**) and (**5**) in mixture which were not further separated, the trithiepin (**12**) and the tetrathiocin (**13**). Compounds (**12**) and (**13**) were then again subjected to flash-chromatography, but the trithiepin (**12**) remained impure of the thiophene (**5**) and the tetrathiocin (**13**) of the dithiin (**6**). The further purification was achieved by treatment these compounds (**12**) and (**13**) with solvents such as pentane, hexane and cyclohexane at room temperature under prolonged stirring.

Phenyl(7-phenyl-1,2,3-trithiepin-4-yl)methanone (**12**): amorphous light-yellow powder (0.4 g, 3%), mp 119-122 °C (Anal. Calcd for $C_{17}H_{12}OS_3$: C, 62.16; H, 3.68; S, 29.28. Found: C, 62.20; H, 3.65; S, 29.27); λ_{max} (CH_2Cl_2) 446s, 336 and 260 mμ; ν_{max} (KBr) 1625 cm^{-1}; δ_H ($CDCl_3$) 7.34-7.45 (m, 4H), 7.48-7.53 (m, 2H), 7.57-7.62 (m, 2H), 7.67-7.71 (m, 2H), 7.86-7.89 (m, 2H); δ_C ($CDCl_3$) 123.81, 126.33, 128.39, 129.06, 129.12, 132.12, 133.33, 135.85, 138.13, 142.27, 153.20, 187.95; MS: m/z 328 $(M)^+$, 296 $(M - S)^+$, 264 $(M - 2S)^+$, 105 $(PhCO)^+$, 77.

Phenyl(8-phenyl-1,2,3,4-tetrathiocin-5-yl)methanone (**13**): amorphous yellow powder (0.84 g, 6.0 %), mp 85 °C (Anal. Calcd for $C_{17}H_{12}OS_4$: C, 56.64; H, 3.35; S, 35.57. Found: C, 56.63; H, 3.39; S, 35.54); λ_{max} (CH_2Cl_2) 448, 428, 332 and 258 mμ; ν_{max} (KBr) 1637 cm^{-1}; δ_H ($CDCl_3$) 7.17-7.42 (m, 5H), 7.44-7.58 (m, 4H) 7.59-7.64 (m, 1H), 7.66-7.83 (m, 2H); δ_C ($CDCl_3$) 128.54, 128.65, 129.08, 129.19, 129.28, 129.34, 129.44, 131.82, 132.58, 137.20, 138.29, 140.04, 141.44, 154.43, 186.96; MS: m/z 360 $(M)^+$,328 (M –

S)$^+$, 296 (M – 2S)$^+$, 264 (M – 3S)$^+$, 105 (PhCO)$^+$, 77.

Conversion of the tetrathiocin (13) into the trithiepin (12). a) *With trimethyl phosphite.* A solution of the tetrathiocin (**13**) (0.1 g, 0.27 mmol) in TMP (5 mL) was allowed to stand at rt for 12 h to give trithiepin (**12**) (50 %). b) *By pyrolysis.* A solution of the tetrathiocin (**13**) (0.1 g, 0.27 mmol) in toluene (5 ml) was refluxed for 5 h to give the trithiepin (**12**) (40 %).

Reaction of tetrathiocin (13) with DMAD. A solution of tetrathiocin (**13**) (0.36 g, 1 mmol) and DMAD (0.3 g, 2.5 mmol) in benzene (10 mL) was refluxed for 2 h. After cooling, the reaction mixture was chromatographated to isolate dithiin derivative (**4**) (0.19 g, 60% yield) from the remaining numerous reaction products.

Monitoring of the reaction of cinnamylideneacetophenone (1) with sulfur and TEA in several solvents. Reaction mixtures of cinnamylideneacetophenone (**1**) (0.234 g, 1 mmol), sulfur (0.5 g, 16 mg/at) and TEA (2 mL) in the following solvents (5 mL): ethyl acetate (AcOEt, 1), DMF (2), DMSO (3), MeCN (4), HMPA (5), Py (6), MeNO2 (7), MeCOMe (AcMe, 8), no solvent (9), morpholine (Mo, 10), in this latter case TEA was not added, are allowed to stand under stirring for seven days, during which the appearance and disappearance of typically and differently colored compounds (**2**), (**3**) and (**7**) in the several mixtures were chosen as reference indexes in TLC (cyclohexane/ethyl acetate, 9:1) (Figure 3).

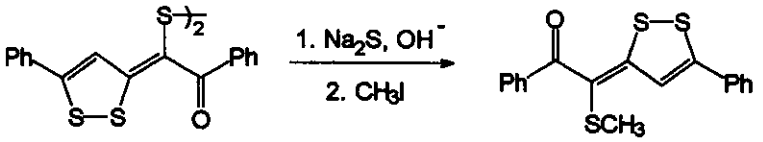
ACKNOWLEDGEMENTS

Authors are grateful to the Italian M.U.R.S.T. for partial financial support.

REFERENCES AND NOTES

1. Part I is to be considered ref. 3.
2. As we have reported in detail in our previous work[3] and in the part of this test relative to "Solvent effect", triethylamine interacts with sulfur in polar aprotic solvents to give a mixture named "activated sulfur" containing cationic and anionic species of sulfur, like the gaseous ammonia.[4]
3. U. Chiacchio, A. Corsaro, A. Rescifina, M. G. Testa, and G. Purrello, *Heterocycles*, 1993, **36**, 223.
4. T. L. Peppard and J. A. Elwidge, *Chem. And Ind.*, 1979, 552; J. A. Elwidge, S. P. Jones, and T. L. Peppard, *J. Chem. Soc., Perkin Trans. I*, 1982, 1089.

5. Literature[6] reports the formation of a similar methylthio derivative (**11**) starting from **10**, which is an isomer of 7.



10 **11**

6. G. Caillard and Y. Mollier, *Bull. Soc. Chim. Fr.*, 1972, 151.
7. P. D. Bartlett and T. Ghosh, *J. Org. Chem.*, 1987, **52**, 4; R. D. Bacchler, J. P. Hummel, and K. Mislow, *J. Am. Chem. Soc.*, 1973, **95**, 4442; U. Anthoni, C. Christophersen, N. Jacobse, and A. Svendsen, *Tetrahedron*, 1982, **38**, 2425.
8. A final structural elucidation must await for a X-Ray analysis.
9. M. Schmidt and U. Görl, *Angew. Chem.,Int. Ed. Engl.*, 1987, **26**, 887
10. Really, in HMPA cinnamylidenacetophenone (**1**) and sulfur react in a minimum extent after 48 h, while using morpholine as basic agent and solvent the reaction course is very fast.
11. CrC Handbook of Chemistry and Physics, D. R. Lide and H. P. R. Frederikse eds, 75th Edn, CRC Press, London, 1994.
12. M. Scholtz, *Ber.*, 1895, **28**, 1730.
13. D. D. Perrin, W. L. F. Armarego, and D. F. Perrin, "Purification of Laboratory Chemicals", 2nd Edn., Pergamon Press, New York, 1980.

Received, 11th August, 1997