Novel and Efficient Method for the synthesis of Cyclic Trithiocarbonates employing Cs_2Co_3/CS_2 system

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Abstract
A novel and efficient method for the synthesis of the cyclic trithiocarbonates from various alkyl dihalides has been developed employing $CsCO_3/CS_2$ system in the presence of DMSO. This method is cleaner, greener and the yield of the reaction is much better than the other available methods.

Keywords: Dibromo alkane, Carbon disulfide, Cesium carbonate, Cyclic trithiocarbonates.

Introduction

Organic trithiocarbonates are significant compounds having numerous applications in polymerization as reversible augmentation chain transfer agents,[11,14] and agrochemicals for pest and insect control.[9,17] The trithiocarbonates have also been reported to have very good anticancer activity[34] and they have been used in the synthesis of bioengineering functional copolymers which have been found to possess anti-tumor activities against cervical cancer cells. HDAC (Histone deacetylase) inhibitors are novel cancer preventing agents[10] which are vital for epigenetic or non-epigenetic regulation, for arrest of cell cycle in cancer cells and apoptosis causing death. Recently, their role has been taken into cognizance and test on cancer patients has been validated. Various HDAC inhibitors are undergoing clinical trials for use as anti-cancer drugs.

As a result, the FDA has approved the use of two HDAC inhibitors, vorinostat and depsipeptide. Anticancer drugs have also been proposed for fabrication of organic/inorganic nanohybrids[4] by using trithiocarbonate as a modifier reported as chemotherapy drug for breast cancer via their interactions with the DNA macromolecules of cancer cells. Recently, trithiocarbonates have also been reported to have antimicrobial properties.[23]

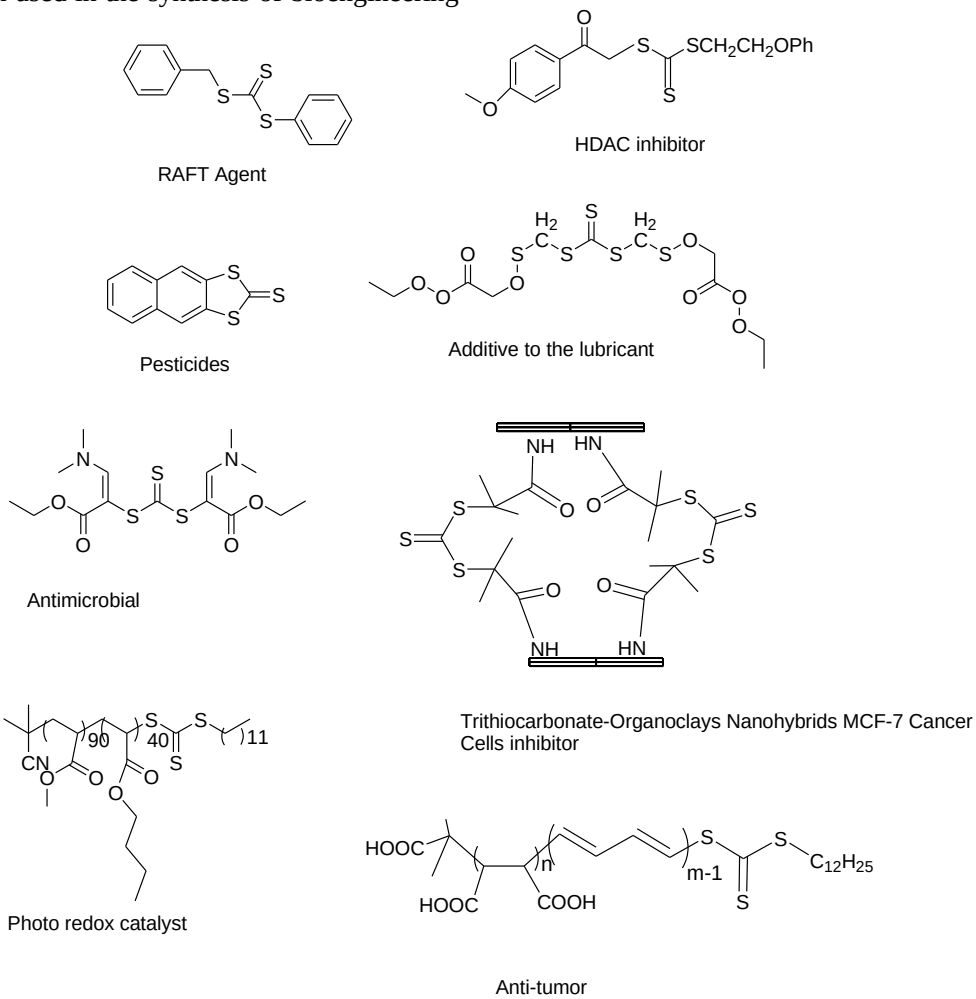


Figure 1: Biologically potent structurally diverse trithiocarbonate derivatives

Trithiocatbonates have also been employed as intermediates in synthesis of thioacetates,[18] dithiocarboxylate derivatives,[32] thiols,[24] trithiocarbonates S-oxides,[13] ketenes,[21] sulfines,[25] nitro benzothiazole[33] and property enhancing agents used in lubrication.[1] The trithiocarbonates have been reported as electrolyte additive[5] for lithium-ion batteries and photoredox catalysts[14] for radical polymerization.

The emerging importance of the trithiocarbonate compounds in various fields has motivated researchers to work for their better and easier synthesis and explore various other forms of trithiocarbonates. The traditional methods for the synthesis of trithiocarbonate used chlorodithioformates,[16] thiophosgenes,[12] reaction between CS_2 and alkyl halides under harsh conditions[28-31] and reacting episulphide[8] or epioxides[29] with metal xanthates.

In the synthesis of the trithiocarbonate, the most important step is the generation of the trithiocarbonate anion (CS_3^-) in the reaction which has been done by the different researcher in several ways from CS_2. Here basic conditions have been employed through anion exchange resin,[35] K_2CO_3,[2] K_3PO_4,[26] KF/Al_2O_3,[27] basic Al_2O_3,[19] NH_4OH,[37] KOH[22] and NaOH.[20] The extreme conditions have been used by the researchers in these processes for the generation of the CS_3^- ion. Moreover, the time of the reaction is longer and the yields are also less. So, there is need to improve the time and yield with easier and simpler process without using extreme conditions.

Earlier, we reported simple protocol for S-alkyl carbamates, O, S-dialkyl dithiocarbonates (xanthates), carbamates, carbonates and dithiocarbamates from a variety of starting materials using the Mitsunobu reagent and Triton-B.[7] Recently we have reported method for synthesis of symmetrical trithiocarbonate[36] in the presence of Cs_2CO_3 and Triton-B. In this communication, we reported synthesis of another significant class of trithiocarbonates namely cyclic trithiocarbonate making the use of Cs_2CO_3/CS_2 system.

The reported method is effective as it involves cheaper and less toxic reagents than the other available methods.[3,30] Moreover, the reaction in this method occurred at room temperature and so it is energy saver too. The work-up and recovery of the compound are simple and easy as compared to the available methods.

Material and Methods

The reaction was carried out with the reagents manufactured by Sigma Aldrich and Merck. Products formed in the reaction were recognized by comparing spectral and physical studies with the compounds available in the literature. IR spectra of the compounds were done on Bomem MB-FTIR machine. NMR spectra were done at 400 MHz on Bruker Advance spectrometer in the solvent $CDCl_3$ and DMSO with TMS as standard. The elements in the compounds were analyzed by Carlo-Erba EA 1110 analyzer and it matched the calculated values. Structure of all the products was ascertained using analytical data thus obtained.

Representative Procedure for the [1,3] Dithiolane-2-thione: A round bottom flask was charged with 2.0 mL of DMSO which was further added with 2.0 mmol of CS_2 and 2.0 mmol of Cs_2CO_3 with constant stirring for 20 min. The reaction mixture turned red in color. After this, it was further charged with 2.0 mmol of 1,2 dibromo ethane and stirred for 3.0 hrs. The qualitative and quantitative assessments of the product were ascertained by the thin layer chromatography. After the end of the reaction, the mixture was emptied in distilled water and the organic compound was extracted with ethyl acetate (20x3) mL which was further obtained in pure form by column chromatography using n-hexane and ethyl acetate (in the ratio1:9) as eluent.

Concentration of the compound on rotatory evaporator gave yellowish oil with yield 94%. The spectral characterization of the synthesized cyclic trithiocarbonates was verified by the previously synthesized cyclic trithiocarbonates available in the literature (3a-3f).[3,30] The compounds 3g and 3h are new and were characterized by the IR, ^{1}H NMR, ^{13}C NMR and elemental analysis.

Selected spectral data for representative compounds are:

i) **1,3-Dithiolane-2-thione: 3a,** yellow oil; elemental analysis of $C_3H_4S_3$ calculated (found)%: C, 26.22(26.02); H, 2.95(2.89); S, 70.59(70.47); IR (cm^{-1}): 1063 (C=S); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 45.0, 229.8; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 3.97 (s, H).

ii) **1,3-Dithiane-2-thione: 3b,** yellow oil; elemental analysis of $C_4H_6S_3$ calculated (found)%: C,31.87(31.53); H, 4.29(4.26); S, 63.82(63.25); IR (cm^{-1}): 1063 (C=S);^{13}C NMR (100 MHz, $CDCl_3$) δ =21.6, 35.5, 221.9; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 2.45 (m, 2H), 3.24 (t, 4H).

iii) **1,3-Dithiepane-2-thione: 3c,** yellowish oil; elemental analysis of $C_5H_8S_3$ calculated (found)%: C, 36.54 (36.28); H 4.906(4.78); S, 58.54(58.32); IR (cm^{-1}): 1063 (C=S); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 30.2, 33.7, 208; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 2.87 (m, 4H), 1.96(m, 4H).

iv) **5-Methyl-[1,3]dithiane-2-thione: 3d,** yellow oil; elemental analysis of $C_5H_8S_3$ calculated (found)%: C, 36.54(36.21); H, 4.906(4.89); S, 58.54(57.97); IR (cm^{-1}): 1063; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 16.9, 37.4, 43.1, 208; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.03(3H doublet), 2.21(1H multiplet), 2.83(4H doublet).

v) **5,5-Dimethyl-[1,3]dithiane-2-thione: 3e**, yellow oil; elemental analysis of $C_6H_{10}S_3$ calculated (found)%: C, 40.40(40.08); H, 5.65(5.53); S, 53.94(54.02); IR (cm^{-1}): 1063; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 24.4, 34.9, 49.0, 208; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.09 (6H singlet), 2.79 (4H singlet).

vi) **4-Methyl-[1,3]dithiolane-2-thione: 3f,** pale yellow oil; elemental analysis of $C_4H_6S_3$ calculated (found)%: C, 31.87(31.69); H, 4.29(4.27); S, 63.82(63.53) IR (cm^{-1}):

1063; ^{13}C NMR (100 MHz, $CDCl_3$) δ =19.1, 48.9, 48.7, 208.0; ^{1}H NMR (400 MHz, $CDCl_3$): δ =1.39, 3.90, 4.1.

vii) **4,4-Dimethyl-[1,3]dithiolane-2-thione: 3g,** yellow oil; elemental analysis of $C_4H_6S_3$ calculated (found)%: C, 31.87(31.72); H, 4.29(4.23); S, 63.82(63.49); IR (cm^{-1}): 1063; ^{13}C NMR (100 MHz, $CDCl_3$): δ =26.9, 52.0, 56.7, 208; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.46(6H singlet), 3.86(2H singlet).

viii) **4,5-Dimethyl-[1,3]dithiolane-2-thione: 3h,** yellow oil; elemental analysis of $C_5H_8S_3$ calculated (found)%: C, 36.54(36.33); H, 4.906(4.86); S, 58.54(58.28); IR (cm^{-1}): 1063; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 17.4,53.3, 208; ^{1}H NMR (400 MHz, $CDCl_3$): δ= 1.41 (6H singlet), 4.10 (2H doublet).

Results and Discussion

Keeping in mind the base sensitivity of trithiocarbonate ion and our focus on mild reaction conditions (room temperature and normal atmospheric conditions), we took equimolar amount of CS_2 and Cs_2CO_3 in DMSO. This mixture was stirred for 20 min which resulted in the formation of trithiocarbonate ion[37] indicated by the change of the color of the mixture from colorless to dark red. This solution was further added with 2.0 mmol of dibromide (1, 2- dibromo ethane) with constant stirring (scheme 1) which gave yellow colored solution.

The reaction was monitored periodically with TLC for the formation of product. The yellow oily product formed after 3.0 hrs was identified through analytical and spectral analysis.

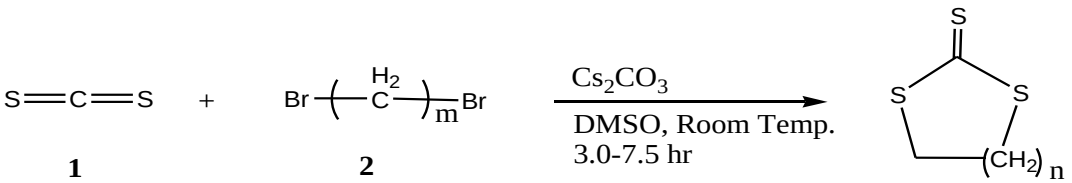
The IR spectra of the compound showed stretching at 1063 cm^{-1} which was due to C=S. ^{13}C NMR of the compound showed peaks at δ = 45.0 due to C-C and peak at δ = 229.8 due to C=S. The ^{1}H NMR showed singlet peak at δ = 3.98 which tells that all protons are equivalent. Thus, the structure of 3a was confirmed as 1,3-Dithiolane-2-thione.

The reaction was then optimized for the solvent, time of the reaction and amount of the phase transfer catalyst. The optimization process was done by taking 2.0 mmol of dibromide (1, 2- dibromo ethane) and 2.0 mmol of CS_2 in the absence of solvent and in the presence of several solvents like dimethyl sulfoxide (DMSO), dimethyl formamide (DMF), water (H_2O) and toluene at room temperature. The yield of the reaction was maximum in the DMSO which was thus optimized as solvent for the reaction. The yield of the reaction with different solvents is given in table 1.

Moving ahead, the amount of the Cs_2CO_3 was optimized and we found that 2 equivalents of the mild base (Cs_2CO_3) were able to give 94% of the yield while if the amount of the Cs_2CO_3 was decreased to 1.5 and 1.0 equivalent, the yield also decreased to 80 and 60% respectively, thus the yield was found to be directly proportional to the concentration of the mild base used in the reaction.

After optimizing the process, several cyclic trithiocarbonates were synthesized by reacting dialkyl halides with CS_2 in DMSO using Cs_2CO_3 as catalyst. The process gave cyclic trithiocarbonates (3a-3h) in excellent yield at normal temperature and pressure which are given in the table 2.

The process worked well for the cyclic trithiocarbonates with lesser strain. The strained trithiocarbonates were formed with lesser yield. Moreover, the substitution in the dibromides with electron withdrawing groups decreased the yield while electron donating groups increased the yield of the reaction. The steric effect also played the role in the yield of the reaction. We found that higher is the overcrowding at the carbon containing the Br group, lesser is the yield of the reaction.



1 **2**

where n = m-1

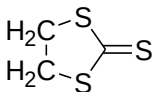
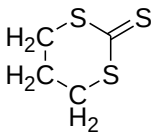
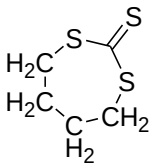
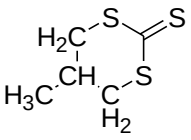
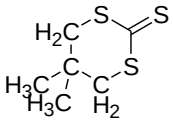
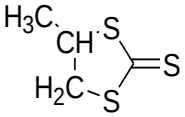
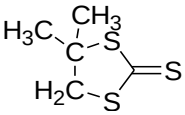
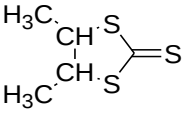
3

Scheme 1: Synthesis of cyclic trithiocarbonates

Table 1
The yield of the reaction with different solvents

S.N.	Solvent	Time of the reaction hrs	% Yield
1	Absent	No reaction	Nil
2	DMSO	3.5	94
3	DMF	4.8	45
4	H_2O	No reaction	Nil
5	Toluene	No reaction	Nil

Table 2
Cyclic trithiocarbonates (3a-3h) in excellent yield at normal temperature and pressure

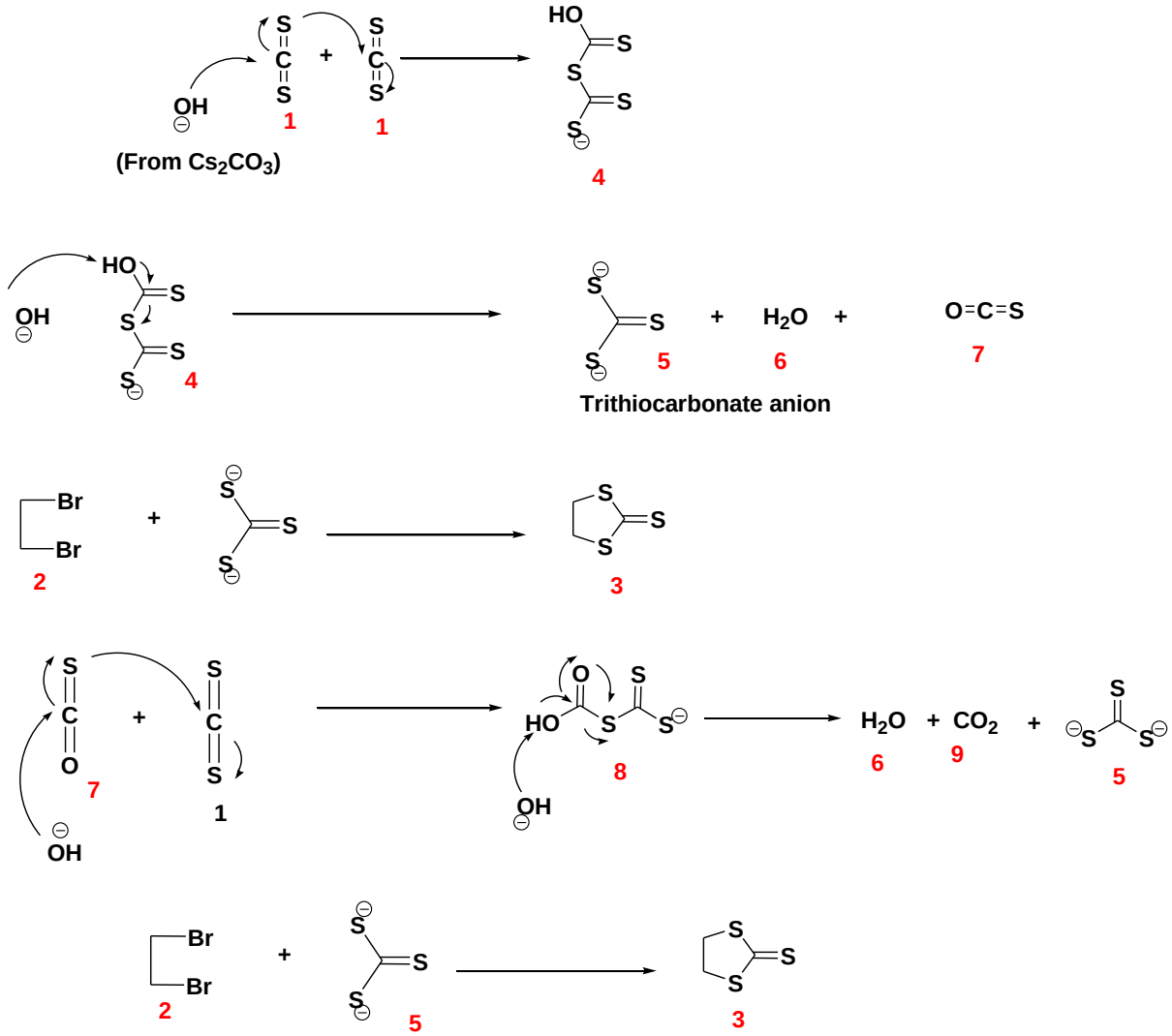
Compound No.	Dibromo alkane	Product	Time in hrs	% Yield
3a	Br- CH_2-CH_2-Br[3]		3.5	94
3b	Br-CH_2-CH_2-CH_2-Br[30]		3.0	90
3c	Br- CH_2- CH_2- CH_2-CH_2-Br[3]		3.8	92
3d	Br-CH_2-CH(CH_3)-CH_2Br[30]		5.0	88
3e	Br-CH_2-C(CH_3)$_2$$CH_2$Br[30]		6.0	80
3f	Br- CH(CH_3)-CH_2-Br[30]		5.2	88
3g	CH_3-C(CH_3)(Br)-CH_2-Br		3.0	84
3h	Br-CH(CH_3)-CH(CH_3)-Br		3.0	80

Possible mechanism of the reaction may be given as in scheme 2. The carbon atom in the CS_2 is highly electrophilic in nature and the S^- is highly nucleophilic which favors the formation of the intermediate 4. The intermediate formed in the reaction is highly acidic and is stabilized by the Cs_2CO_3 which acts as an inorganic base.

Conclusion

In conclusion, a new method is being reported for the synthesis of cyclic trithiocarbonates in the presence of triton-B at normal conditions of temperature and pressure. This process is a cheaper, cleaner and greener process than the available methods. All reagents used in the synthesis are either easily available or they can be easily synthesized in laboratory.

The process gives higher to excellent yields with easy and simple workup. The process saves energy as it occurs at room temperature and saves time which are the best features of the developed protocol.



Scheme 2: Proposed mechanism of the reaction

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(Received 24th January 2021, accepted 02nd April 2021)