

Review Article

The Synthetic Chemistry of Carbon Monosulfide

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Dedicated to Professor Henning Lund on the occasion of his 70th birthday.

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On the basis of an extensive on-line literature search a short review (with 50 references) is given of the preparative-scale synthetic chemistry of carbon monosulfide.

Contrary to popular belief carbon monosulfide, CS, in spite of its instability, is readily available for preparative work on a molar scale.¹ It constitutes, in favorable cases, a convenient building block which allows the preparation of a considerable variety of thiocarbonyl and related compounds. Even Nature, in interstellar space, emulates the activities of the experimental chemist and generates more complex molecules from abundant carbon monosulfide.^{1–4}

The present review is based on an exhaustive on-line search of the relevant literature. Carbon monosulfide is the subject of extensive work in theoretical chemistry (cf. Ref. 1; for latest developments cf. Refs. 5–9) as well as physical, especially laser, chemistry (cf. Ref. 1; for latest developments cf. Refs. 10–16), which is beyond the scope of this review as is the vast literature on the observations of CS in space (cf. Ref. 1; for latest developments cf. Ref. 4). Another subject outside the scope of this review is, with a single exception, metal complexes with CS ligands, since these complexes are generally prepared in ways not involving CS, nor do they serve as synthetic sources of CS.¹

The generation of CS

Most of the synthetic work on record has been carried out at -78°C in a high vacuum (<0.1 Torr) line with CS generated by a high-voltage (≈ 10 kV) a.c. discharge through CS₂ gas (*vide infra*).¹ A similar procedure where thiophene replaces the CS₂ suffers from the disadvantage of concurrently generated hyper-reactive thio-ketene.^{1,17,18} The breakthrough from micro- to macro-

scale preparations was achieved when a robust and high-yielding vacuum line set-up was devised in Klabunde's laboratory.^{1,19} At the trap temperatures required to freeze out efficiently the admixed CS₂, potentially explosive solid CS is deposited, thus the presence of CS₂ during the reactions must be tolerated. Attempts to generate CS by aromatization assisted thermal extrusion from cyclic precursors have not yet met with success.^{20,21}

Reactions of CS

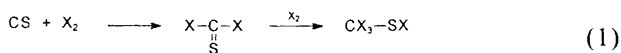
The dimerization of CS and related reactions. Low-temperature work with matrix-isolated species has revealed the formation of the dimer C₂S₂ in its triplet ground state by reaction of ¹CS with ³CS. C₃S₂ and S(CS)₂ are also formed under these conditions.²² While better preparative methods are available for the isolable C₃S₂, neither C₂S₂ nor S(CS)₂, though spectroscopically characterized, are of preparative interest because of their extreme instability and the inefficiency of their preparation.

The polymerization of CS. The smooth polymerization of gaseous CS and the explosive polymerization of solid CS are the most conspicuous properties of CS which must be taken into account in any preparative work involving CS.¹ The formation of the dark colored (CS)_n wherever CS is present in the absence of suitable reaction partners is tantamount to an indicator effect for the presence of such reactants and also a persistent nuisance with regard to the cleaning of the glassware. Although the polymer (CS)_n can readily be prepared in quantity its full characterization has not been achieved yet. Recent work²³ strongly suggests that '(CS)_n' might actually be a

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mixture of the better known polymers $(CS_2)_n$ and $(C_3S_2)_n$. Recently prepared or theoretically analyzed 'oligomers' of CS, i.e. the C_4S_4 ²⁴ species cyclobutanetetrathione, the C_6S_6 ^{25,26} species 7,8-dithiabicyclo[4.2.0]oct-1(6)-ene-2,3,4,5-tetrathione, 3,4,9,10-tetrathiatricyclo[6.2.0^{2,5}]deca-1(8),2(5)-diene-6,7-dithione, 4,5,9,10-tetrathiatricyclo[6.2.0^{3,6}]deca-1(8),3(6)-diene-2,7-dithione, cyclohexanehexathione, and 3,4,7,8,11,12-hexathiatetracyclo[8.2.0.0^{6,9}]dodeca-1,5,9-triene, the C_8S_8 ²⁷ species 3*H*,6*H*-[1,2]dithiolo[3'',4'':4',5']thieno[3',2':4,5]-thieno[3,2-*c*]-1,2-dithiole-3,6-dithione and 1,2,3,4,5,6,7,8-octathiadicyclopent[*cd*,*ij*]-*s*-indacene, and the C_9S_9 ²⁶ species benzo[1,2-*d*:3,4-*d'*:5,6-*d''*]tris[1,3]dithioletrithione and 3*H*,6*H*,9*H*-benzo[1,2-*c*:3,4-*c'*:5,6-*c''*]tris[1,2]dithiole-3,6,9-trithione are apparently chemically unrelated to monomeric CS and the polymers formed by CS.

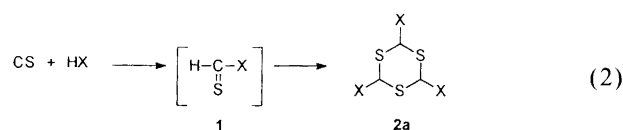
Reactions of CS with elements. The violent reaction of CS with molecular oxygen is a hazard which must be dealt with in any preparative work involving CS.¹ Its reaction with oxygen atoms is the basis of well-established laser chemistry.¹ Obviously, the reaction of CS with elemental sulfur to form CS_2 would be without preparative interest and has so far not even warranted examination from a mechanistic point of view. This reaction has, however, been inferred from its behavior *vis-à-vis* the heavier chalcogens (*vide infra*).²⁸ The reaction of CS with selenium and tellurium mirrors leads to $CSSe$ and $CSTe$, respectively, which have been identified spectroscopically, but no preparative versions of these reactions are on record.²⁸ Chlorine, bromine, and iodine react with CS at room temperature to form the corresponding thiocarbonyl halides CSX_2 , eqn. (1), which were frozen out for spectroscopic examination after 8 cm migration in the vacuum line, corresponding to 10 ms.



In later preparative work along these lines Klabunde *et al.* examined these same reactions as well as the corresponding reactions of the interhalogens $BrCl$ and ICl . The corresponding thiocarbonyl halides were undoubtedly formed, but immediately reacted further with the halogen or interhalogen. Thus, cocondensation of CS with chlorine gave trichloromethanesulfonyl chloride (CCl_3SCl), with bromine tribromomethanesulfonyl bromide (CBr_3SBr), with bromine chloride trichloromethanesulfonyl chloride, tribromomethanesulfonyl bromide, and bromdichloromethanesulfonyl chloride ($CBrCl_2SCl$), and with iodine chloride trichloromethanesulfonyl chloride in substantial yields. No GLC-stable products survived the reaction between CS and iodine.^{19,29} Inconclusive, essentially negative, results were obtained upon codeposition of the pseudohalogen cyanogen bromide and CS.²⁹ Codeposition of nickel vapor and CS at 10 K led to an

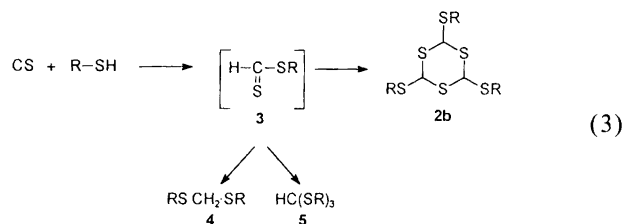
ill-defined solid matrix which yielded spectroscopic evidence of the presence of minute amounts of nickel-tetrakis(thiocarbonyl), $Ni(CS)_4$. The authors assume that the $Ni(CS)_4$ might well be produced by pyrolysis of the ill-defined primary products in the mass spectrometer rather than by direct reaction between CS and elemental nickel.³⁰ Apparently, this work provides few, if any, clues of preparative relevance. Subsequent painstaking work by White²⁹ failed to detect any reaction between nickel metal and CS (nor between CS and nickel tetracarbonyl).

Reactions of CS with hydrogen halides. Hydrogen chloride and HBr, respectively, add to CS to form the corresponding thioformyl halides **1**, which immediately trimerize to the corresponding 2,4,6-trihalo-1,3,5-trithianes **2a**, eqn. (2).^{19,29}



Reaction of CS with CO. Low-temperature work with matrix isolated species has revealed a photoreaction between these two species which leads to thioxoethenone $O=C=C=S$ (earlier gas-phase work had already demonstrated the existence of this species), a highly labile species which could be characterized spectroscopically.³¹ Thioxoethenone and the reactions leading to it are, so far, without preparative significance.

Insertion of CS into sulfur-hydrogen bonds. The reaction between thiols and CS is obscured by uncharted secondary reactions. Three thiols have been examined, eqn. (3), cf. Table 1.



The inference of a dithioformate **3** as the primary product is by no means straightforward since it would be expected to trimerize to the corresponding 1,3,5-trithiane **2b** rather than enter into the unprecedented carbophilic addition of a thiol to a thiocarbonyl group

Table 1. The attempted thiocarbonylation of thiols with CS.

Thiol	Product(s) (yield, %)	Ref.
BuSH	BuSCH ₂ SBU 4a (18), (BuS) ₃ CH 5a (13)	32
PhCH ₂ SH	PhCH ₂ SCH ₂ SCH ₂ Ph 4b (13), (PhCH ₂ S) ₃ CH 5b (4)	32
PhSH	PhSCH ₂ SPh 4c (27)	32

as required by the mechanism suggested to account for the formation of the observed secondary products shown in Table 1.³²

Insertion of CS into sulfur–chlorine bonds. The most fruitful pursuit in preparative CS solution chemistry so far has been its insertion into the sulfur–chlorine bond of sulfonyl chlorides RSCl and of chlorodisulfanes RSSCl, eqns. (4) and (5), cf. Tables 2–4. Thus, disulfur dichloride neatly inserts two molecules of CS to form dithiobis(thiocarbonyl chloride) **8** in an impressive 50% yield while benzenesulfonyl chloride and CS yield 73%

Table 2. The thiocarbonylation of sulfonyl chlorides RSCl.

RSCl	Yield of RSCSCL 6 (%)	Ref.
CCl ₃ SCl	75	34,36
MeCOSCl	Unstable	37
CCl ₃ SCCl ₂ SCl	49	34,38
(EtOOC) ₂ CClSCl	16	39,40
4-MeC ₆ H ₄ SO ₂ (PhS)CClSCl	No reaction!	40
PhSCl	73	33–35

Table 3. The thiocarbonylation of chlorodisulfanes ('thiosulfonyl chlorides') RSSCl.

RSSCl	Yield of RSSCSCI 7 (%)	Ref.
CICOSSCl	53	34,41
CCl ₃ SSCl	57	34,41
CH ₃ COSSCl	50	34,41
CCl ₃ CCl ₂ SSCl	19	34,41
(EtOOC) ₂ CClSSCl	Moderate, not determined	39,40

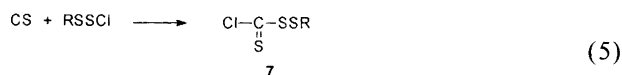
Table 4. The thiocarbonylation of oligofunctional compounds X(SCl)_n.

Starting material	Products (yield, %)	Ref.
CISSCl	CICSSSCSCI 8 (50)	33–35
(EtOOC) ₂ C(SCl) ₂	(EtOOC) ₂ C(SCSCL) ₂ (not isolated), (EtOOC) ₂ C(Cl)SSSC(COOEt) ₂ SCl (not isolated), (EtOOC) ₂ CS ₅ (not isolated)	40,42
(PhNHCO) ₂ C(SCl) ₂	Not identified	40,42
CISCH ₂ CH ₂ SCl	CISCH ₂ CH ₂ SCSCL (not isolated), CICSSCH ₂ CH ₂ SCSCL (not isolated)	43,44
CISCF ₂ CF ₂ SCl	CICSSCF ₂ CF ₂ SCSCL (38)	43,44
CISCl ₂ SCF ₂ CF ₂ SCCl ₂ SCl	CICSSCl ₂ SCF ₂ CF ₂ SCCl ₂ SCSCL (not isolated)	44
<i>cis/trans</i> -1,3-Dichloro-2,2,4,4-tetramethylcyclobutane-1,3-disulfonyl dichloride	Two mono- and two di-insertion products (not isolated)	40
1,3,4-Thiadiazole-2,5-disulfonyl dichloride	CICSS–C ₂ N ₂ S–SCSCL (100)	43,44
1,3,5-Triazine-2,4,6-trisulfonyl trichloride	The mono- and the di-insertion product, both too unstable for isolation	43,44

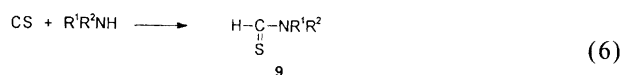
Table 5. The thioformylation of primary and secondary amines with CS.

Amine	Product(s) (yield, %)	Ref.
<i>tert</i> -Octylamine	<i>N-tert</i> -Octylthioformamide 9a (0.5)	32
Benzylamine	<i>N</i> -Benzylthioformamide 9b (1.3)	32
Morpholine	4-Thioformylmorpholine 9c (49)	17,18,32
Piperazine	1-Thioformylpiperazine 9d (30), 1,4-bis(thioformyl)piperazine 9e (40)	32
Aniline	<i>N,N</i> -Diphenylformamidine 10 (2–6)	32
Diphenylamine	No reaction	32

of phenyl chlorodithioformate.^{33–35} A wide range of compounds containing S–Cl bonds react smoothly with CS, almost regardless of their structure. However, recently a singular case of a sulfonyl chloride was found which does not react with CS, cf. Table 2.



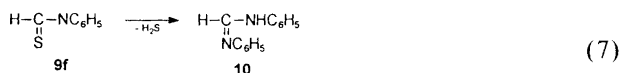
Insertion of CS into nitrogen–hydrogen bonds. Primary and secondary amines insert CS into an NH bond to form the corresponding thioformamides **9**, eqn. (6), cf. Table 5 (no reaction was observed with the essentially non-basic and non-nucleophilic diphenylamine).



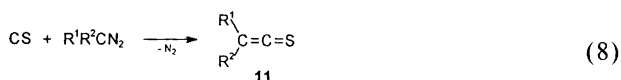
However, the yields are erratic and more efficient procedures are available for the synthesis of these reaction products. The small number of cases examined does not invite any summary conclusions from the observed yields. One should, however, note the extremely mild reaction conditions which might be beneficial in the case of amines with, say, base-sensitive groups.

The formamidine derivative obtained with aniline could be envisaged to be formed by condensation of

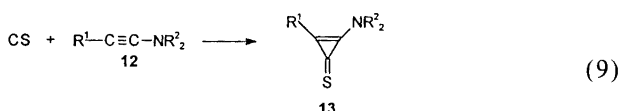
thioformanilide **9f** (the expected insertion product) with aniline, eqn. (7). However, this postulated condensation (with loss of hydrogen sulfide) has never been demonstrated to proceed between authentic thioformanilide and aniline.



Reactions of CS with ylides. The first reaction of this kind to be observed was that between codeposited bis(trifluoromethyl)diazomethane and CS and led to a minute yield of the isolable bis(trifluoromethyl)thioketene **11a**, eqn. (8), cf. Table 6.²⁹ Later experiments carried out in solution on a preparative scale led to substantially improved yields in two cases where isolable thioketenes **11** were formed, but failed in cases where labile thioketenes were expected to form.⁴⁵ Resonance-stabilized diazoalkanes, such as diazomalonic ester, apparently do not react with CS.⁴⁴ Benzyl azide, phenyl azide, 9-fluorenyl azide, and trimethylsilyl azide do not react with CS.⁴⁴ Dimethylsulfonyl(ethoxycarbonyl)methanide does react with CS, but no well-defined products could be isolated.⁴⁴ 4,4-Dimethyl-2,5-dioxo-1-(phenyliodonio)cyclohexan-1-ide and 2,2-dimethyl-4,6-dioxo-5-(phenyliodonio)-1,3-dioxan-5-ide do not react with CS.⁴⁴



Cycloadditions of CS to nucleophilic alkynes. In spite of the fact that the nucleophilic alkynes **12** react with the CS₂ unavoidably present when CS is generated, respectable yields of the corresponding cyclopropenethiones **13** could be achieved in this reaction, eqn. (9), Table 7.



Abstraction reactions with CS. Early work by White²⁹ demonstrated the ability of CS to convert cyclohexene oxide into cyclohexene with concomitant formation of

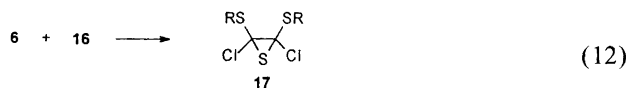
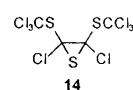
Table 6. Reactions between diazoalkanes and CS.

Diazoalkane	Product (yield, %)	Ref.
(<i>t</i> -Bu) ₂ CN ₂	(<i>t</i> -Bu) ₂ C=C=S 11b (30)	45
(CF ₃) ₂ CN ₂	(CF ₃) ₂ C=C=S 11a (0.14)	29
S(CH ₂ CMe ₂) ₂ CN ₂	S(CH ₂ CMe ₂) ₂ C=C=S 11c (42)	45
N ₂ C(COOEt) ₂	No reaction	44
CH(N ₂)COOEt	N ₂ released, no reaction products isolated	45
Ph ₂ CN ₂	N ₂ released, no reaction products isolated	45
9-Diazofluorene	N ₂ released, no reaction products isolated	44

Table 7. Reactions between 1-aminoalkynes and CS.

1-Aminoalkyne 12	Yield (%) of cyclopropenethione 13	Ref.
1-(Diethylamino)propyne 12c	24	46
1,2-Bis(dimethylamino)ethyne 12a	67	46
1,2-Bis(diethylamino)ethyne 12b	32	46

carbonyl sulfide (yield 9%). The observation of 2,3-bis(trichloromethylthio)-2,3-dichlorothiirane **14** as a by-product of the thiocarbonylation of trichloromethanesulfonyl chloride (*vide supra*) was interpreted as resulting from the following sequence of events: attack of CS on the chlorodithioformate **6**, loss of carbon disulfide from the α-dithiolactone **15** thus formed leading to a carbene **16**, which in turn attacks the primary



product **6**, eqn. (12).^{34,36} However, attempts to develop this into a general method for the generation of carbenes from thiocarbonyl compounds and CS under extremely mild conditions failed although there was evidence that thiobenzophenone is indeed attacked and desulfurized by CS.⁴⁴

Reactions of metal coordinated CS. It has been shown that a platinum bound thiocarbonyl ligand of a palladiaplathinathirane or -selenirane can take part in a ring expansion to the corresponding diheterathietanethione or -selenetanethione.⁴⁷

Compounds which fail to react with CS. An extensive list of compounds inert to CS can be found in Ref. 1.

Reactions where CS has been shown not to be involved in spite of earlier claims to the contrary. The complex secondary reactions observed in the electrochemical reduction of CS₂, eventually leading to 4,5-dimercapto-1,3-dithiole-2-thione derivatives have been shown in a painstaking investigation not to include steps involving CS.⁴⁸

Conclusions

With standard vacuum line equipment large scale preparative work with CS is a practical proposition. Three factors limit the usefulness of this methodology: (a) the unavoidable presence of CS₂ which can lead to undesired by-products, (b) the requirement of low pressure which complicates work with volatile reaction partners, (c) the facile polymerization of CS which precludes slow reactions of CS with the chosen reaction partners. By and large, the known preparative chemistry of CS parallels that of the analogous isocyanides⁴⁹ rather than that of the apparently less analogous CO. The quantum chemical characterization of CS as a σ donor and π acceptor⁵⁰ explains the experimental observation that it reacts with both electrophilic (such as sulfenyl chlorides) and nucleophilic reaction partners (such as 1-aminoalkynes). For experimental details and further interesting reactions of the products obtained from the above-mentioned substrates and CS the cited literature should be consulted.

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