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REVIEW ARTICLE

Reactions of carbon disulfide with N-nucleophiles

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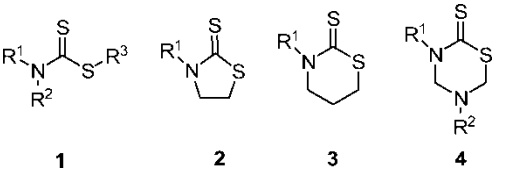
The aim of this review is to present reactions of carbon disulfide with N-nucleophiles which form nitrogen-carbon bonds. Compounds with an amino or imino group react, in the presence of a base, with carbon disulfide to give dithiocarbamates. They can be converted to esters on treatment with an alkylation reagent. Acidification affords dithiocarbamic acids. Dithiocarbazates are formed on treatment of hydrazines with carbon disulfide. A lot of dithiocarboxylation products are valuable building blocks for heterocycles.

Keywords: Carbon disulfide; Dithiocarbamate; Dithioimidocarbonate; Dithiocarbazate; Heterocyclization

1. Introduction

The majority of the reactions between carbon disulfide and N-nucleophiles involve addition of carbon disulfide to N−H bonds. The products of these reactions, dithiocarbamate salts, can be transformed into dithiocarbamic acids and esters which find application in the synthesis of a wide range of organosulfur compounds.



1 **2** **3** **4**

S-Alkyl dithiocarbamates of the general structure **1** including cyclic derivatives **2–4** show antibacterial [1–4], anthelmintic [5, 6], fungicidal [1, 2, 4, 7–10], herbicidal [7, 11] antifouling [12], growth depressant [13], and algicidal activity [14]. They are also effective catalysts for photopolymerization [15] and vulcanization [16, 17]. Their chelating properties allow them to be used as antidotes against nickel and copper poisoning, in analytic determination of heavy metals and in wastewater treatment. They can also affect metal-containing enzymes [18].

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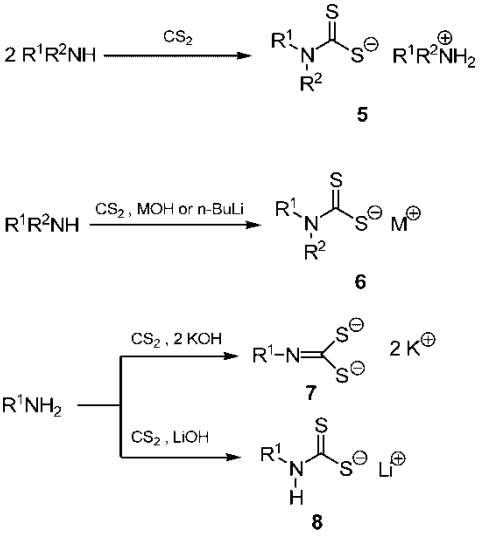
In the field of medicine these compounds are used in the treatment of chronic alcoholism and in fungi- and bacteria-related diseases, and they have also received some attention as potential auxiliaries in oncological chemotherapy and in the prevention of arteriosclerosis [19]. Dithiocarbamates exhibit low chronic and acute toxicity in humans and mammals, although their high reactivity, associated with their chelating properties and their high affinities for HS-containing proteins, is responsible for adverse effects including neurotoxicity, antithyroid properties, eye and skin sensitization [20].

2. Reactions with amino compounds

2.1 *Reactions with ammonia, primary and secondary amines*

Ammonia [21], primary [22] and secondary aliphatic amines [23] react with carbon disulfide in acetone or ethanol to afford dithiocarbamate salts **5**. This reaction is one of the earliest known reactions in organosulfur chemistry and was originally investigated by Hofmann more than 130 years ago [24]. Metal dithiocarbamates **6** (M=Na, K, Li) are obtained by treating primary and secondary amines with carbon disulfide in the presence of NaOH, KOH [25–28] or n-BuLi [29]. This method has the advantage that only one equivalent of amine is required, and consequently it is more efficient than the original method when expensive amines are employed as substrates. Reactions between carbon disulfide and exocyclic amino groups of 2-aminobenzothiazoles performed with KOH and with LiOH selectively afforded potassium dithioimidocarbonates **7** and lithium dithiocarbamates **8**, respectively [30].

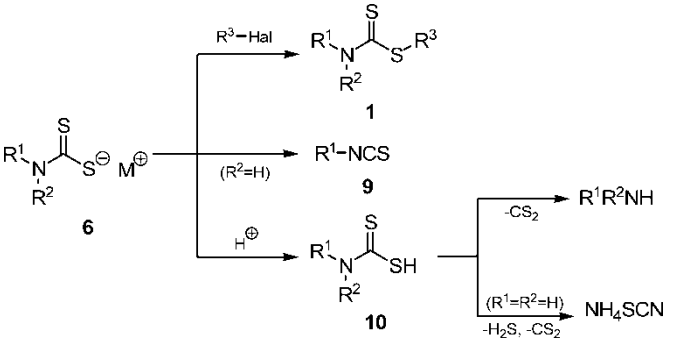


Ammonium and trialkylammonium salts **6** [M: NH_4^+, $(R^3)_3NH^+$] are synthetically useful and may be prepared by the reaction of carbon disulfide and an amine in dry ethanol or diethylether with excess ammonia or trialkylamine [31–33].

Heavy metal dithiocarbamates can be obtained by adding of a soluble salt of a heavy metal to the solution of a sodium or ammonium salt of the required dithiocarbamic acid [34–36]. Aromatic amines (especially diarylamines) react less readily with carbon disulfide, and dithiocarbamate salts from these substrates are best prepared under anhydrous conditions using strong bases such as NaH in THF [37] or KOH in DMSO [38].

The mechanism for the formation of dithiocarbamate salts has been investigated, and the rate of formation is inversely proportional to pH [39–42].

Dithiocarbamate salts have variable stability. Purification is achieved by crystallization but the yields can be very disappointing since the salts may decompose, particularly upon heating. N-Alkyldithiocarbamate salts **6** (R^2=H) are unstable [43, 44] and in basic solution decompose to yield isothiocyanates **9**. A procedure for the preparation of isothiocyanates **9** from primary amines and carbon disulfide by using hydrogen peroxide as the dehydrosulfurization reagent was reported [45]. The early methods developed for this transformation involve heating heavy metal dithiocarbamate salts (Hg [46], Cu [47], Pb [48, 49], Fe(II) [50, 51]). The use of mercury(II)chloride in acetone [46, 52] is specially suitable for the synthesis of isothiocyanates from esters of amino acids, and for the preparation of isothiocyanates containing basic substituents [53, 54].



Treatment of dithiocarbamate salts **6** with mineral acids produces dithiocarbamic acids **10**. Unsubstituted dithiocarbamic acid **10** (R^1=R^2=H) is obtained by treating ammonium salt with hydrochloric or sulfuric acid at low temperature [21, 34]. The acid is unstable and readily decomposes to give ammonium thiocyanate, carbon disulfide and hydrogen sulfide. Substituted dithiocarbamic acids **10** are generally unstable and readily dissociate to afford amines and carbon disulfide in aqueous solution [34, 36]. Stability appears to increase with increasing pH [55] and depends on the substituents attached to the nitrogen atom. N,N-Dialkyldithiocarbamate salts **6** are generally stable in basic solution but revert under acidic conditions to the starting amine and carbon disulfide [56]. Diphenyldithiocarbamic acid **10** ($R^1 = R^2 = Ph$) is relatively stable and can be recrystallized from benzene [57].

Alkali metal and ammonium salts **6** can be alkylated with haloalkanes to yield S-alkyl dithiocarbamates **1** [25, 26, 36, 58–97]. A highly efficient and simple synthesis of **1** is based on the one-pot reaction of amines, carbon disulfide, and alkyl halides without using a catalyst under solvent-free conditions [98].

Phenoxarsin-10-yl dithiocarbamates were prepared by the reaction of 10-chlorophenoxarsine and sodium dithiocarbamates [99] .

Tributyltin dithiocarbamates were obtained directly from the corresponding amines by reaction of carbon disulfide in the presence of bis(tributyltin)oxide [100].

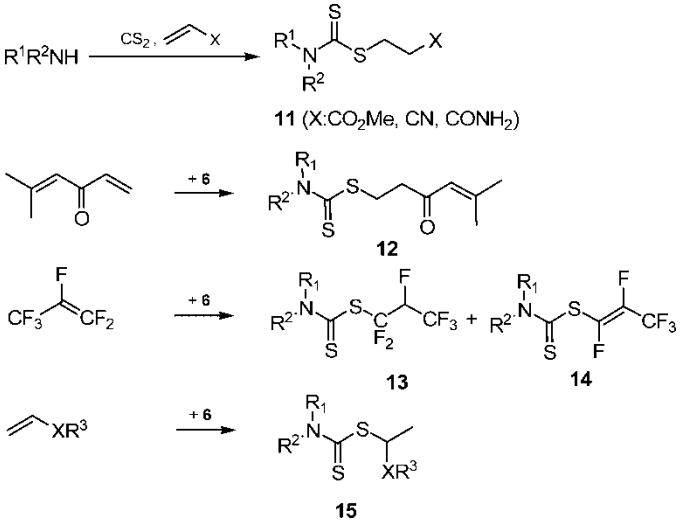
The esters **1** may also be prepared by one-pot methods without isolation of the intermediate dithiocarbamate salt **6**, since the latter is rapidly alkylated at room temperature [101–104]. The presence of cesium carbonate and tetrabutylammonium iodide facilitated efficient reaction of primary and secondary amines with carbon disulfide and alkyl halides. The method is mild and chemoselective [105]. Different kinds of dithiocarbamates **1** were prepared by one-pot procedure from primary and secondary amines, carbon disulfide and a variety of alkyl halides or epoxides in the presence of anhydrous potassium phosphate [106–110].

A three component coupling was performed to combine diamines and amino alcohols with carbon disulfide and halides in the presence of a cesium base and tetrabutylammonium iodide, leading to the synthesis of mono dithiocarbamates [111].

Partially protected amino carbohydrates can be converted into dithiocarbamates without affecting free hydroxyl groups [112]. In addition to haloalkanes, other alkylating agents such as dihaloalkanes [113–116], nitrates [117], and β-lactones [118] can be employed. Reduction of alkyl N,N-dimethyldithiocarbamates has been used preparatively for the synthesis of thiols [119]. S-Aryl-N,N-diisopropyldithiocarbamates **1** yield aromatic thiols on alkaline hydrolysis [120]. Aminomethyl esters are accessible from the reaction between dithiocarbamate salts and amines in the presence of formaldehyde [82, 121].

S-Aryl dithiocarbamates **1** are obtained when dithiocarbamate salts **6** are treated with aromatic hydrocarbons containing labile halo or nitro substituents [122–124], or with reactive halohetarenes [75]. Arylation of dithiocarbamate salts can also be achieved with diazonium [59, 125, 126] or diaryliodonium salts [127].

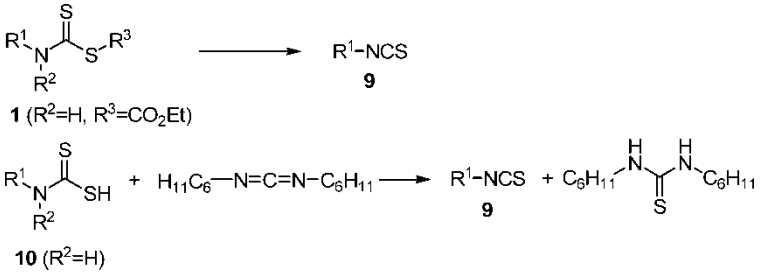
Dithiocarbamic acids **10** and their salts **6** can be added to activated carbon−carbon multiple bonds to give S-alkyl or S-alkenyl dithiocarbamates **1** [59, 128–144]. Highly efficient one-pot reactions of a wide range of aliphatic, aromatic, primary, secondary, and hindered amines with carbon disulfide and α,β-unsaturated compounds were carried out in water or in methanol (in the presence of anhydrous potassium phosphate) to give dithiocarbamates **11** [145, 146]. Addition of dithiocarbamate salts **6** [133, 134] to activated alkenes containing more than one C=C takes place at the less hindered double bond to yield **12**.



Treatment of dithiocarbamate salts **6** with perfluoropropene leads to both the S-alkyl ester **13** and the S-alkenyl ester **14** [147]. Dithiocarbamate salts **6,** prepared *in situ* from carbon disulfide and secondary amines, are added to the α-carbon atom of vinyl ethers and thioethers to afford **15** [148, 149].

Dithiocarbamate salts **6** can be added to non-activated double bonds only at high pressures. Thus, cyclohexene reacts with carbon disulfide and secondary amines to yield **1**

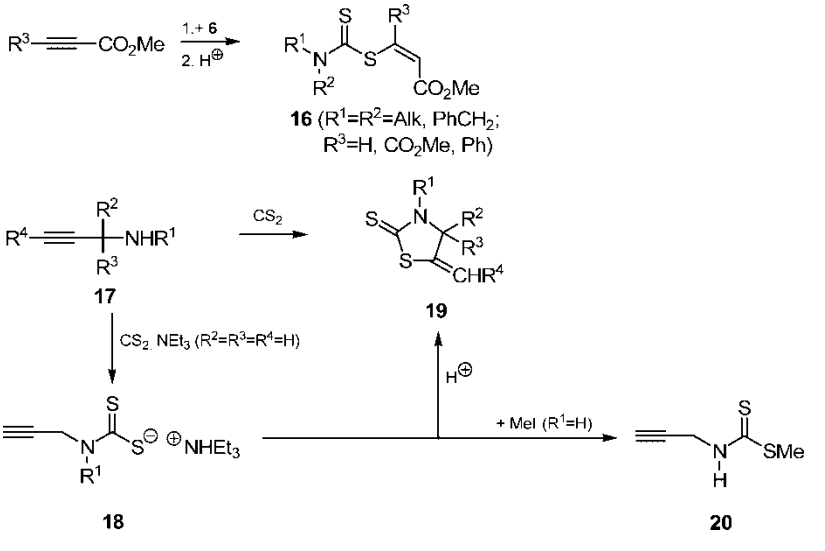
(R^3 = cyclohexyl) [150].



Ethoxycarbonyldithiocarbamate esters **1** (R^2=H, R^3=CO_2Et) (prepared from **6** and ethyl chloroformate) may be decomposed to yield isothiocyanates **9** in good yields [151–155] .

Additions of dithiocarbamic acids to C=N bonds are more complicated. For example, reaction of **10** (R^2=H) and dicyclohexyl carbodiimide leads to an unstable addition compound which decomposes to give isothiocyanate **9** and dicyclohexyl thiourea [156, 157].

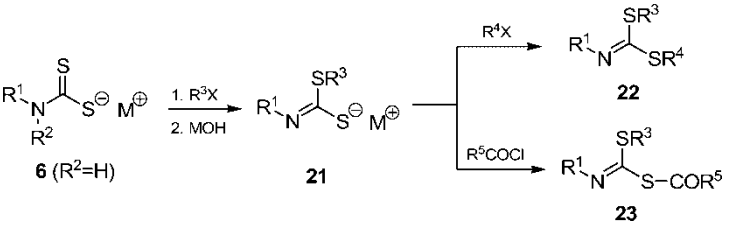
S-Alkenyl esters **16** can be obtained by the addition of dithiocarbamate salts **6** to carbon-carbon triple bonds. Reactions involving both alkynes and diynes have been described, and addition may proceed regio- and/or stereospecifically [158–171].



When propargylamines **17** are treated with a mixture of triethylamine and carbon disulfide, the dithiocarbamate salts **18** are produced [172]. Cyclization of **18** in aq. HCl gives methylenethiazolidinethiones **19** [173–175]. Reaction with methyl iodide leads to methyl dithiocarbamate **20** [176–180].

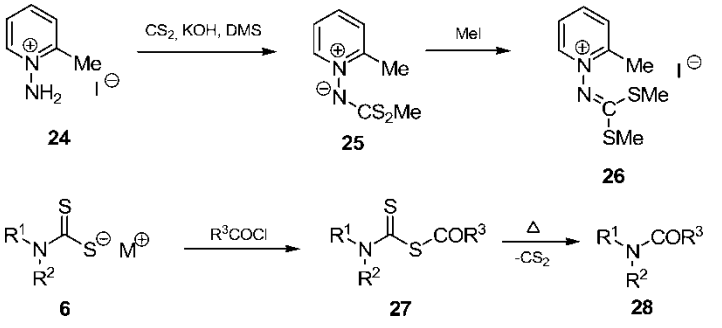
Monoalkylation of dithiocarbamate salts **6** (R^2=H) yields intermediate **21** which can be further alkylated to give dialkyl dithioimidocarbonates **22**. As in the case of dithiocarbamic acid esters **1** prior isolation of **6** is unnecessary and the reaction may be carried out in one-pot from a primary amine, carbon disulfide and an alkylating agent [181–201]. Acylation of the

intermediate **21** affords S-acyl derivatives **23** [202, 203].



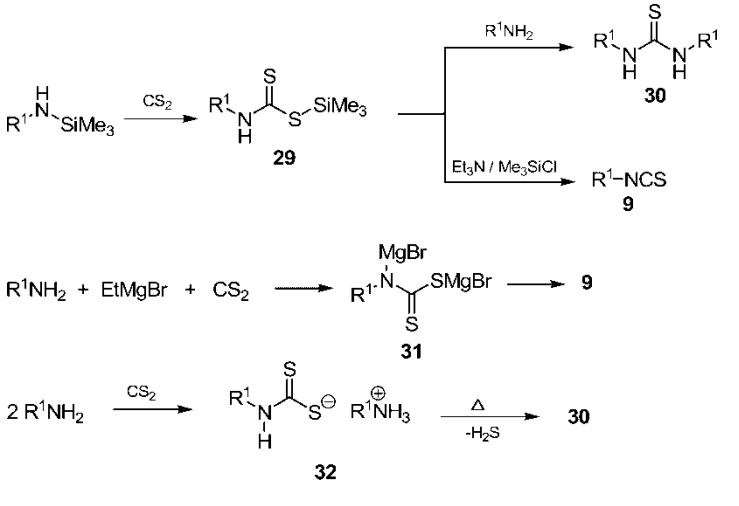
Treatment of 1-aminopyridinium salts **24** with carbon disulfide, KOH and dimethyl sulfate produces **25** which can be alkylated to give **26** [204] .

S-Acyldithiocarbamates **27** are obtained by acylation of dithiocarbamate salts **6** with acid chlorides [87, 205–207]. These mixed anhydrides are unstable and lose carbon disulfide on heating to afford amides **28** [205].



Bis-(S-acyldithiocarbamates) result when dithiocarbamate salts are treated with phosgene [208]. S-Acylation of **6** by chlorides of thioacids [209], carbamoyl chlorides [210] and chlorides of acid hydrazides [206] have also been described. Polymer-bound S-acyldithiocarbamates have been used as recyclable selective acylating agents [211].

Trimethylsilyl amines react with carbon disulfide to produce S-silyl dithiocarbamates which decompose above 100 °C to regenerate the starting materials [212, 213]. Treatment of N-silylated primary amines with carbon disulfide leads to similar dithiocarbamates **29** which can be decomposed to afford thioureas **30** or isothiocyanates **9** [214, 215].



The thermal decomposition of intermediates **31** prepared *in situ* from primary amines, a Grignard reagent and carbon disulfide, gave aryl and alkyl isothiocyanates **9** [216]. Such reactions are best carried out in three stages: (a) monometallation with Grignard reagent, (b) reaction with carbon disulfide and (c) addition of the second equivalent of Grignard reagent [217]. A similar procedure can also be used when the metallating agent is butyl lithium.

When a hot solution of a primary amine (in pyridine, water or ethanol) is treated with carbon disulfide the intermediate dithiocarbamate salt **32** loses hydrogen sulfide and symmetrical thiourea **30** is produced [218–222]. The process is catalyzed by different reagents such as sulfur [223] and dimethylchloroformiminium chloride (prepared *in situ* from DMF and thionyl chloride) [224]. Furthermore, the addition of hydrogen peroxide [223] or NaOH [225, 226] to remove hydrogen sulfide formed in the reaction greatly increases the rate of thiourea formation. A basic ZnO/Al_2O_3 composite can afford an efficient catalyst in the synthesis of symmetric N,N'-disubstituted thioureas **30** from primary amines and carbon disulfide [227]. The use of a solid base easily prepared by anchoring a guanidine to mesoporous silica as efficient catalyst for the preparation of thioureas from primary amines and carbon disulfide was reported [228].

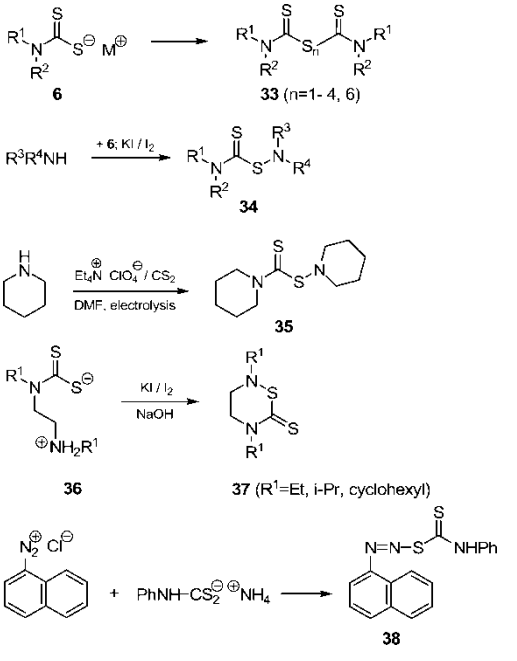
Primary aliphatic amines can be converted *in situ* to alkylammonium dithiocarbamate salts which decompose in boiling ethanol to yield thioureas [22, 221, 229]. Optically active symmetrical thioureas have been obtained from optically active amines and carbon disulfide in toluene [230].

Unsymmetrically substituted N,N′-dialkylthioureas can be obtained under phase transfer conditions in a one-pot reaction by the treatment of an amine with carbon disulfide and reaction of the intermediate dithiocarbamate salt with a second amine [231].

Peptide coupling reagents can be used as versatile reagents for the formation of aliphatic isothiocyanates and thioureas on solid phase from carbon disulfide and the solid-phase anchored aliphatic primary amines [232].

With a few exceptions, secondary amines do not react with carbon disulfide to afford symmetrical tetrasubstituted thioureas [233, 234].

Dithiocarbamate salts **6** can be oxidatively coupled with a variety of mild oxidizing agents to yield thiuram disulfides **33** (n=2) [235–248].



On an industrial scale the conversion can be accomplished using chlorine [249] or oxygen in the presence of copper acetate [250]. Dithiocarbamate salts of azoles react analogously [251, 252]. Mixed thiuram disulfides result when mixtures of dithiocarbamate salts are treated with ammonium persulfate [253]. When oxidation is carried out in the presence of cyanide ion, the reaction product is the monosulfide **33** (n=1) [254–257]. Activation of dithiocarbamate salts with 2-halo-3-alkyl-4-phenylthiazolium salts also gave thiuram monosulfides under very mild conditions [258].

Unsymmetrical thiuram monosulfides can be obtained by the reaction of water soluble **6** with (dialkylthiocarbamoyl)sulfur chloride [259].

Dithiocarbamate salts **6** react with sulfur monochloride and sulfur dichloride to produce thiuram trisulfides **33** (n=3), tetrasulfides (n=4) and hexasulfides (n=6) [25].

Oxidative coupling of **6** with sodium hypochlorite or KI/I_2 in the presence of amines affords thiocarbamoylsulfenamides **34** [260–264].

Unsubstituted sulfenamides **34** (R^3=R^4=H) result when **6** is treated with chloramine [260] or hydroxylamine-O-sulfonic acid [265].

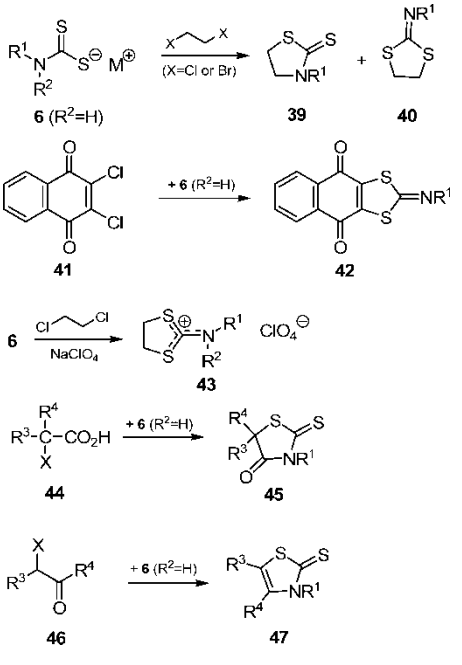
Electrolysis of a mixture of carbon disulfide and piperidine in DMF using tetraethylammonium perchlorate as the supporting electrolyte affords sulfenamide **35** [266].

Oxidation of dithiocarbamate inner salts **36** leads to tetrahydro-1,2,5-thiadiazine-6-thiones **37** [267].

Reaction of 1-naphthyldiazonium salt with ammonium salt of phenyldithiocarbamic acid gives 1-naphthylazophenyldithiocarbamate **38**. Similar reaction has been carried out with several other aryl diazonium salts [268, 269].

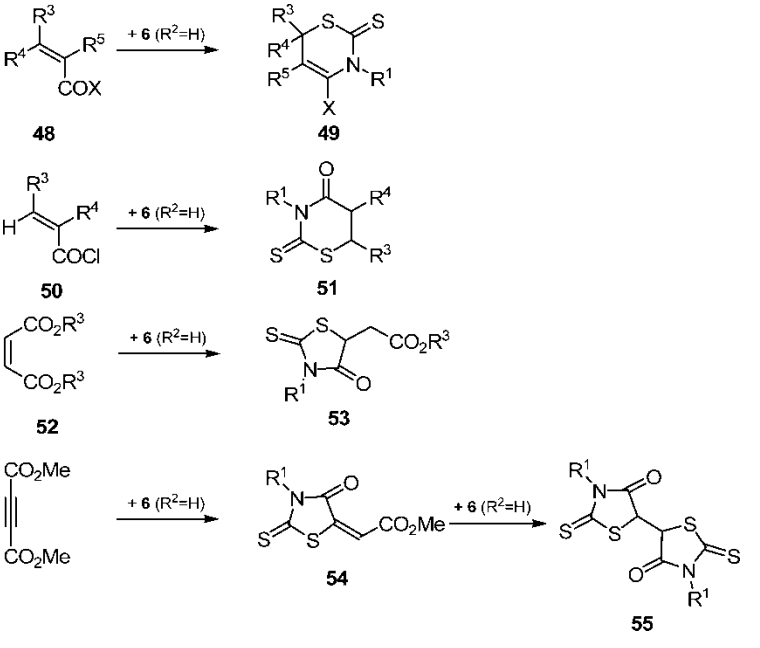
Reaction of dithiocarbamates **6** with reagents containing two reactive functional groups leads to the formation of heterocycles. Reactions may be carried out with both isolated dithiocarbamate salts or mixtures of base, amine and carbon disulfide. Alkylation of **6** (R^2=H) with 1,2-dihaloalkanes affords thiazolidines **39** [270, 271] and 1,3-dithiolanes **40** [272]. Activated dihalides **41** give fused 1,3-dithiolanes **42** [273].

The 2-dialkylamino-1,3-dithiolanylium perchlorate **43** is produced on treatment of **6** with 1,2-dichloroethane in the presence of perchlorate ion [274].



Dithiocarbamate salts **6** (R^2=H) react with α-haloacids (or esters) **44** (X=Cl, Br) to afford 2-thioxothiazolidin-4-ones **45** [275–277], and with α-haloketones **46** (X=Cl or Br) [59, 270, 278–284] or α-thiocyanato-ketones **46** (X=SCN) [282] to give thiazoline-2-thiones **47**. Reaction with halo-acetoacetic esters, halodiketones or halopyruvic esters leads to the formation of carbonyl or carboxyl substituted thiazoline thiones [285–288]. Otherwise, ethyl 3-alkyl-4-hydroxy-2-thioxothiazolidine-4-carboxylates were prepared in excellent yields from the reaction of appropriate primary amines with carbon disulfide and ethyl bromopyruvate in the presence of anhydrous potassium phosphate in DMF [289]. Intermediates from the reactions involving α-halo-ketones and dithiocarbamates have been isolated [280, 283, 290, 291].

Salts **6** react with carbon-carbon multiple bonds to afford heterocycles. For example, α,β-unsaturated ketones **48** (X=alkyl) and acids (X=OH) [129, 292] (as well as β-haloalkanoic acids and β-propiolactones [292, 293]) react with **6** (R^2=H) to yield 3,6-dihydro-2H-1,3-thiazine-2-thiones **49**. Tetrahydro-2-thioxo-1,3-thiazin-4-ones **51** result when **6** (R^2=H) is treated with α,β-unsaturated acid chlorides **50** [294].

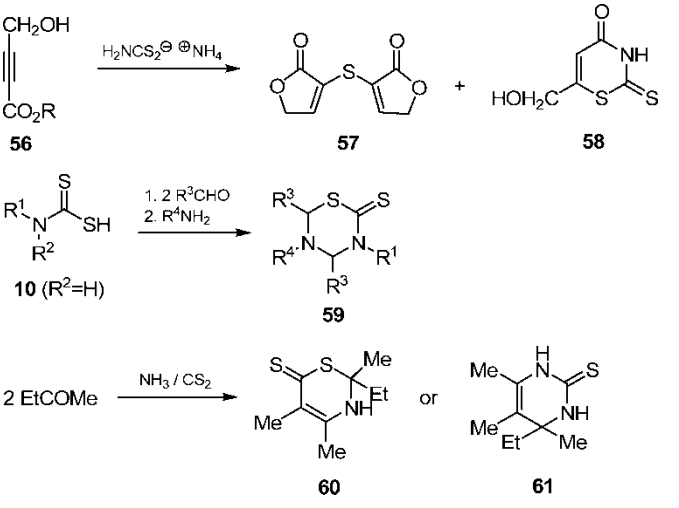


Addition of **6** (R^2=H, M=ammonium) to dialkyl maleates **52** gives 2-thioxo-4-thiazolidinones **53** [295]. Reaction of the same salts with DMAD yields thiazolidines **54** or **55** depending on the reaction conditions [296].

Treatment of 4-hydroxy-2-butynoic acid **56** (R=H) with ammonium dithiocarbamate affords the lactone **57** and the 2,3-dihydro-2-thioxo-4H-1,3-thiazin-4-one **58** (minor product) [160, 161]. By contrast, when **56** (R=Et) is treated similarly, **58** is the major product.

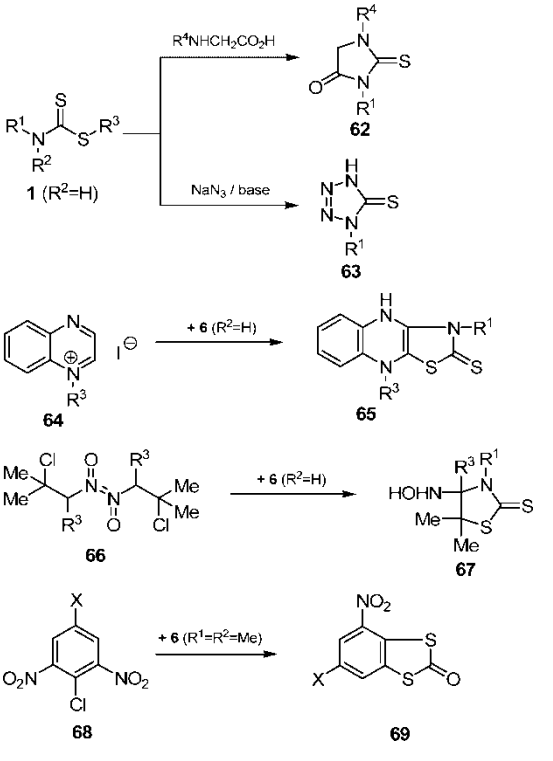
Tetrahydro-1,3,5-thiadiazine-2-thiones **59** are produced when primary dithiocarbamic acid **10** (R^2=H) or a mixture of primary amine and carbon disulfide are treated with alkanals

followed by the addition of an amine [297–301].



Aliphatic ketones react with carbon disulfide and ammonia to yield thiazines or diazines. Below 0 °C, butanone affords the 2,3-dihydro-thiazine-6-thione **60**. At 25 °C the 3,4-dihydro-2(1H)-pyrimidinethione **61** is the reaction product [302, 303].

Nitrogen heterocycles may also be prepared from dithiocarbamic acid esters. Reaction of **1** (R^2=H) with α-amino acids yields imidazolidines **62** [304] and tetrazoles **63** result, via 1,2,3,4-thiatriazoles, when **1** (R^2=H) is treated with sodium azide in alkaline solution [305–307].

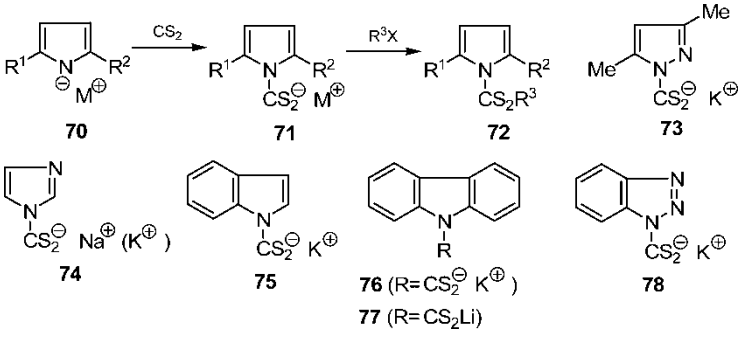


Quinoxalinium salts **64** react with dithiocarbamate salts **6** (R^2=H, R^3=2-thiazolyl or 1,2,4-triazol-3-yl) to yield thiazoloquinoxaline-2-thiones **65** [308]. Reaction of **6** (R^2=H) with dimeric chloronitroso compounds **66** yields thiazolidines **67** [309] and dithiocarbamate salts **6** (R^1=R^2=Me) can be converted into 1,3-benzodithiol-2-ones **69** by reaction with activated arenes **68** (X=nitro or CN) [310, 311].

2.2 *Reactions with azoles*

The salts of N-dithiocarboxylic acids of many azoles (pyrroles, pyrazoles, imidazoles, triazoles and their benzo derivatives) are well characterized. However, the corresponding free acids are unknown, presumably owing to their instability [312].

Reaction of the metallated pyrrole **70** (M=Na, K; R^1, R^2=H, Me) with carbon disulfide in toluene, THF or DMSO affords the extremely hygroscopic and air-sensitive N-dithiocarboxylic acid salts **71** [251, 313, 314]. Salts **71** can be alkylated and arylated in the usual manner to yield esters [251]. Thus, treatment of **71** (M=K, Na; R^1=R^2=H) with iodomethane [251, 315], iodoethane [316] or benzyl chloride [317–319] gives **72** (R^3=Me, Et, benzyl), whilst reaction with benzene diazonium chloride affords the phenyl ester (R^3=Ph) [251]. Esters of the other azoles have been described [315, 320–331].



Addition of carbon disulfide to a suspension of a potassium pyrazolide in benzene or THF leads to potassium pyrazole N-dithiocarboxylate **73** which is moderately stable. Sodium pyrazole-1-dithiocarboxylate is less stable to storage [328, 332].

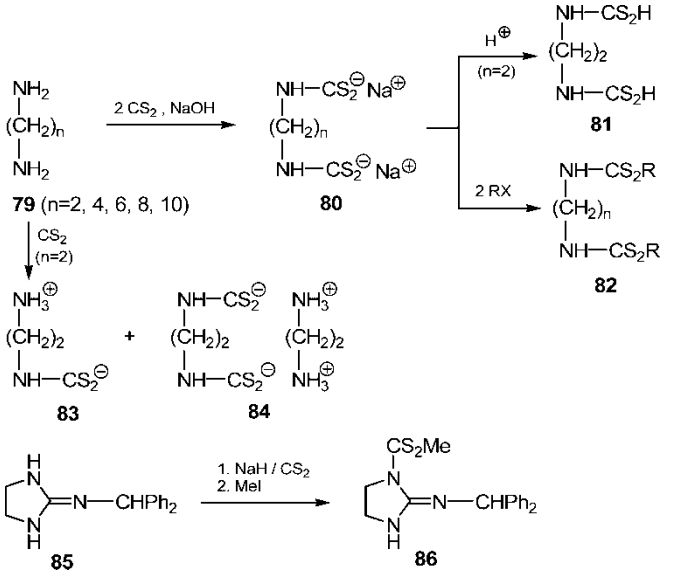
Sodium 1-imidazoledithiocarboxylate **74** was easily prepared from sodium imidazolide and carbon disulfide in ether in nearly quantitative yield [333].

The general method for the preparation of the N-dithiocarboxylic acid salts of imidazole **74,** indole **75** and carbazole **76** consists of treating a solution of the parent amine in dry THF with potassium metal. After 24 h, the mixture is cooled to −78 °C and carbon disulfide added [334]. Lithium carbazole-9-dithiocarboxylate **77** is prepared by treating carbazole with phenyllithium in ether followed by addition of carbon disulfide [320, 321].

Potassium benzotriazole-1-dithiocarboxylate **78** is obtained by the reaction of benzotriazole with potassium hydroxide and carbon disulfide [326].

2.3 *Reactions with diamines*

Primary and secondary aliphatic diamines **79** (n=2, 3, 4, 6, 8, 10) react with carbon disulfide in the presence of NaOH, KOH, LiOH or NH_4OH to yield bis-dithiocarbamate salts **80** [235, 335–341].



Treatment of **80** (n=2) with strong mineral acid affords the bis-dithiocarbamic acid **81** [342]. Bis-dithiocarbamate salts **80** can be alkylated in the usual manner to afford bis-dithiocarbamic acid esters **82**. Reaction of **79** (n=2) with one equivalent of carbon disulfide produces the mono-dithiocarbamic acid **83** (in the form of its betaine) and the bis-dithiocarbamate salt **84** [342, 343]. Mono-dithiocarbamic acids were obtained from other diamines. Reaction of bis-(2-aminoethyl)amine with carbon disulfide yields a water soluble crystalline material which is believed to be the zwitterionic dithiocarbamic acid [344, 345].

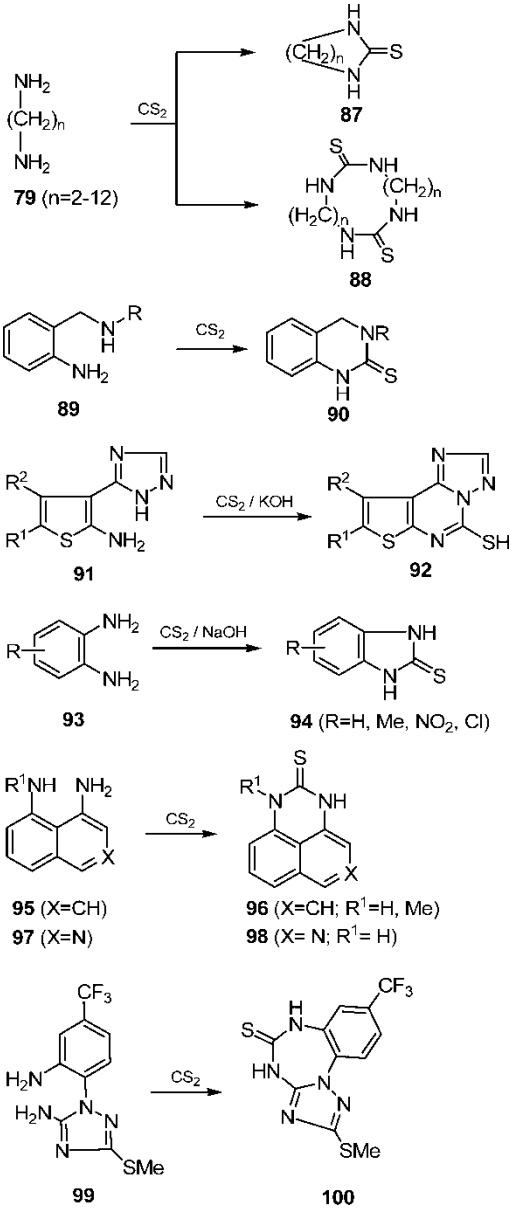
Treatment of the imidazolidine **85** with carbon disulfide and NaH followed by the addition of iodomethane yields the carbodithioic acid ester **86** [346].

Aliphatic diamines **79** (n=2) [275, 347–350], (n=3) [275, 349], (n=4) [349] and (n=6–12) [351–353] react with carbon disulfide in ethanol at 80 °C to give cyclic thioureas **87** and/or **88** depending on the value of n. Thus **79** (n=2-4 and 12) cyclizes to yield only **87** whilst **79** (n=6 and 7) produces **88**. When the substrate is **79** (n=5 or 8) both **87** and **88** are obtained.

Several reactions of aromatic diamines with carbon disulfide have been reported [354–356]. Reaction of 2-aminobenzylamines **89** with carbon disulfide yields 3,4-dihydroquinazoline-2-thiones **90**. The corresponding pyridine derivatives react analogously [357, 358]. Azoles **91** containing appropriately situated amino groups react with carbon disulfide to yield condensed heterocycles **92** [359–361].

Addition of carbon disulfide in hot ethanol/NaOH to aromatic 1,2-diamines **93** generates 1,3-dihydro-2H-benzimidazole-2-thiones **94** [275, 362–367]. Reactions of this type can be carried out using 2-nitroanilines with *in situ* reduction (Raney Ni and hydrazine hydrate or hydrogen sulfide) [368, 369]. *Ortho*-diaminoheterocycles (thiophenes, pyrimidines and pyridines) react with carbon disulfide in pyridine or DMF to yield the corresponding fused

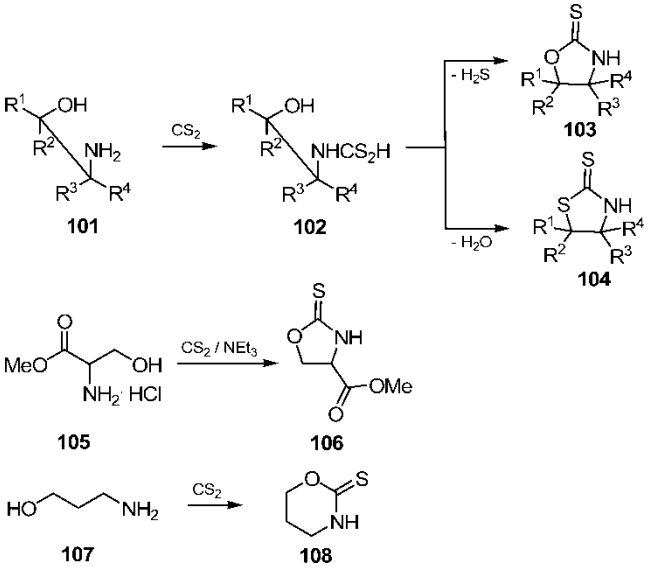
reduced imidazoles [370–375].



1,8-Diaminonaphthalenes **95** are transformed to perimidine-2-thiones **96** on reaction with carbon disulfide [376–379]. Heterocyclic *peri*-diamines react analogously [380]. The isoquinoline **97** reacts with carbon disulfide to yield the pyrido[3,4,5-de]quinazoline-2-thione **98**. Similarly, the 1,2,4-triazole **99** is cyclized to the triazepine **100** when heated with carbon disulfide in pyridine [381].

2.4 *Reactions with amino alcohols and amino phenols*

Treatment of 2-amino alcohols **101** with carbon disulfide leads, initially, to dithiocarbamic acids **102** which cyclize *in situ* to give oxazolidine-2-thiones **103** [382–384], and thiazolidine-2-thiones **104** [385–387]. The product depends on the reaction conditions, and occasionally upon the substituents on the carbon atoms of the amino alcohol. Thiazolidines **104** are obtained when 2-amino primary alcohols are treated with carbon disulfide in basic media [278, 388]. In the absence of alkali, the product is the oxazolidine **103** [389]. Cyclization of the intermediate **102** in the presence of metal salts (especially Pb) favours the formation of **103** [388, 390]. Alkylation of **102** with halo carboxylic acids or their esters followed by treatment with alkali or triethylamine also leads to oxazolidines [391, 392]. If the reaction is conducted in the presence of iodine [393], thiuram sulfides are produced decomposing on heating to afford **103**. In DMSO, in the presence of bases, **103** is obtained in high yield [394].



Chiral oxazolidine-2-thiones **103** (R^1=R^2=R^3=H, R^4=Ph, Bn, i-Pr) are usually prepared from carbon disulfide and amino alcohols either with NEt_3 in CH_2Cl_2 or with an aqueous solution of NaOH or Na_2CO_3 in a biphasic mixture. A cosolvent such as THF or ethanol was sometimes added to facilitate the reaction [395–402] . However, a thermodynamically favoured side product, the corresponding thiazolidine-2-thione **104**, is often formed in substantial quantities regardless of the amount of the carbon disulfide present in the reaction system [399, 401].

A highly practically procedure for preparing the chiral oxazolidine-2-thione auxiliaries **103** using carbon disulfide and the corresponding chiral amino alcohols in the presence of potassium carbonate and hydrogen peroxide is described [403].

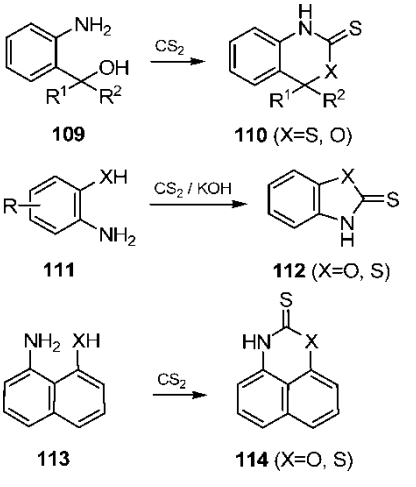
Serine methyl ester hydrochloride **105** reacts with carbon disulfide in THF containing triethylamine to afford the oxazolidine **106** [404].

Tetrahydro-1,3-oxazine-2-thiones **108** are obtained from treatment of 3-amino alcohol **107** with carbon disulfide [393, 405–407]. Alicycle-condensed tetrahydro-1,3(or 3,1)-oxazine-2-thiones are prepared in a similar way [408–414].

Various 1,3-oxazolidine-2-thiones **103** and tetrahydro-1,3-oxazine-2-thiones **108** are prepared by reacting hydrogen peroxide with a mixture of amino alcohols, carbon disulfide and

base in a water-miscible organic solvent. The yields of the heterocyclic products are in the range of 80–100% [415].

2-Amino benzyl alcohols **109** react with carbon disulfide in the presence of base to give 1,4-dihydro-3,1-benzothiazine-2-thiones **110** (X=S) [416–418]. In the absence of base the reaction products are 1,4-dihydrobenzoxazine-2-thiones **110** (X=O) [419–421].



Carbon disulfide reacts with 2-aminophenols **111** (X=O) to yield 2(3H)-benzoxazolethiones **112** (X=O) [364, 422–426]. An efficient method for the preparation of benzoxa(thia)zole-2-thiones **112** is the reaction of aromatic primary amines **111** with carbon disulfide in the presence of catalytic amount of triethylamine followed by treatment with hydrogen peroxide [427].

Heterocyclic amines with *ortho*-hydroxyl groups react analogously [428, 429]. Bicyclic systems with amino and hydroxyl groups in *peri*-positions, as in 8-amino-1-naphthol **113** (X=O), yield fused oxazines **114** (X=O) when treated with carbon disulfide [416].

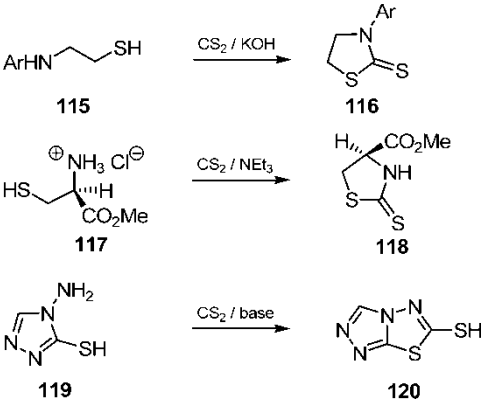
2.5 *Reactions with amino thiols and amino thiophenols*

Reactions between these substrates and carbon disulfide run parallel to those of the previous section [430–432]. Thus, carbon disulfide reacts with aminothiol **115** to afford thiazolidine-2-thione **116**.

L-Cysteine methyl ester hydrochloride **117** reacts with carbon disulfide to yield the 4(R)-isomer of **118** without racemization [404, 433, 434].

Benzothiazoline **112** (X=S) is obtained when 2-amino-thiophenol **111** (X=S) is treated with carbon disulfide [227, 435–437]. Benzothiazolines can also be prepared by heating carbon disulfide with aniline and sulfur, and by the reduction of 2-chloronitrobenzene with hydrogen sulfide in the presence of NaSH and carbon disulfide, or with sodium sulfide (or polysulfide)

in the presence of carbon disulfide [275].

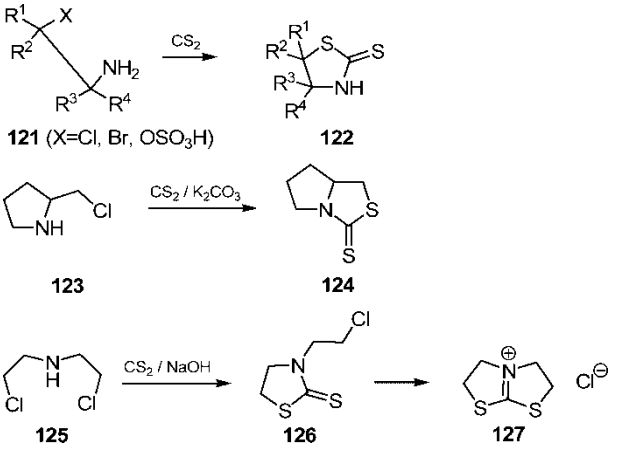


Ring closure of heterocyclic *ortho*-amino-thiols such as pyridines [438–442] and quinolines [443] affords fused thiazolines. Reaction of the s-triazole **119** with carbon disulfide is an effective method for preparing s-triazolo[3,4-b][1,3,4]thiadiazole-6-thiol **120** [444].

8-Amino-1-naphthalenethiol **113** (X=S) was cyclized to condensed thiazine-2(3H)-thione **114** (X=S) [445].

2.6 *Reactions with halo-amines and amino alkylsulfates*

Carbon disulfide reacts with 2-halo-amines [275, 446, 447] or 2-amino-alkylsulfates **121** [290, 448, 449] in basic solution to give thiazolidine-2-thiones **122**.

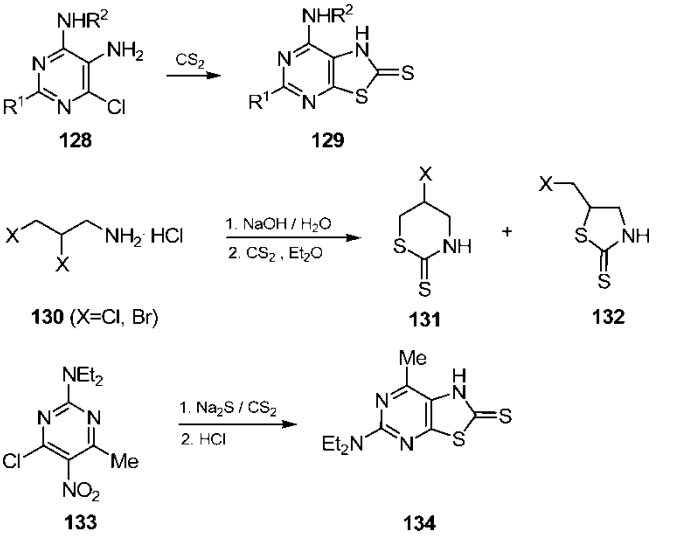


In DMF containing potassium carbonate 2-(chloromethyl)pyrrolidine **123** yields the tetrahydro-1H,3H-pyrrolo[1,2-c]thiazole-3-thione **124** in an analogous manner [450]. Reactions of dihaloalkylamines with carbon disulfide have also been described [451]. Thus, reaction of **125** with carbon disulfide leads, initially, to **126** which cyclizes to afford the tetrahydrothiazolo[2,3-b]thiazolium chloride **127.**

The reaction of heterocyclic amines **128** containing halogen atoms *ortho* to the heteroatom affords bicyclic heterocycles **129** [371]. 2-Chloro-benzylamines react analogously [452].

The reaction of 2,3-dihalo-1-propanamines **130** (X=Cl, Br) with carbon disulfide leads to a mixture (1:1) of 5-halotetrahydro-1,3-thiazine-2-thiones **131** (X=Cl, Br) and 5-(halomethyl)-2-thiazolidinethiones **132** (X=Cl, Br) [453]. Tetrahydro-1,3-thiazine-2-thiones **131** (X=H)

are obtained when aliphatic 3-halo-amines are treated with carbon disulfide in the presence of alkali [454–457].



Thiazolo[5,4-d]pyrimidine-2-thione **134** was obtained from 6-chloro-5-nitropyrimidine **133**, sodium polysulfide and carbon disulfide in one-pot synthesis [458].

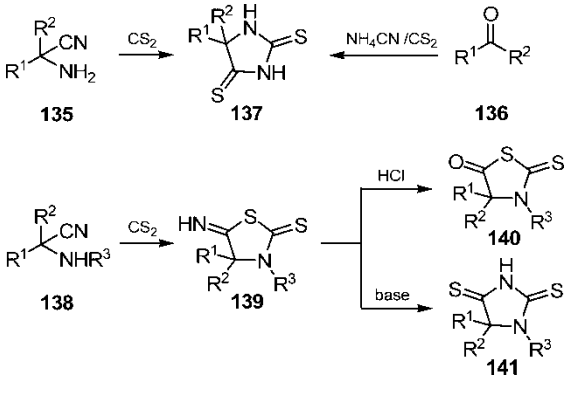
In strongly basic media both 2- and 3-halo-amines afford amino arynes which can be trapped as benzothiazoline **112** (X=S) with carbon disulfide [459].

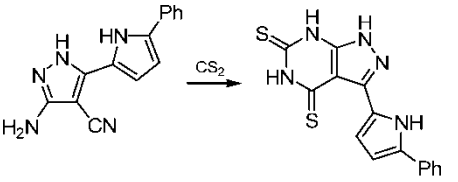
2.7 *Reactions with aminocarboxylic acids and their derivatives*

α-Amino nitriles **135** or mixtures of ketones **136** and ammonium cyanide yield imidazolidine-2,4-dithiones **137** when they react with carbon disulfide [460–463].

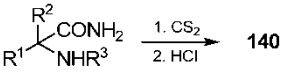
By contrast, N-substituted α-amino nitriles **138** are transformed into 5-imino-2-thiazolidinethiones **139** which afford 2-thioxo-5-thiazolidinones **140** on acidic hydrolysis [464, 465]. Hydrolysis of **139** in basic solution produces 2,4-imidazolidinedithiones **141** [466, see also 467, 468].

The reaction of amino acetonitrile with carbon disulfide has been investigated in detail [462]. The reactivity of N-substituted α-amino nitriles towards carbon disulfide depends on the nature of both the N-substituent and substituents attached to the α-carbon atom [469].





142 **143**



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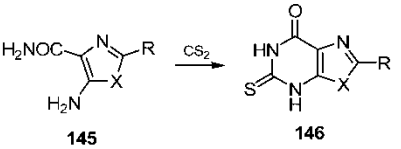
The reaction of aromatic and heterocyclic o-amino nitriles with carbon disulfide in pyridine constitutes a convenient, one-step synthesis of fused pyrimidinedithiones [470–472]. The 3-aminopyrazole-4-carbonitrile **142** and carbon disulfide in pyridine solution afford the fused pyrimidinedithione **143** [473] .

Amides of α-amino carboxylic acids **144** react with carbon disulfide to give 2-thioxo-5-thiazolidinones **140** and ammonia [462, 468, 474].

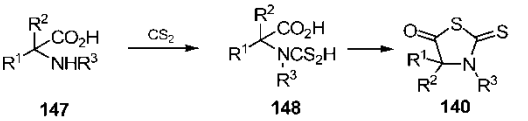
β-Amino carboxylic acid amides **145** (X=S or NMe) react in an entirely different manner to produce **146** and hydrogen sulfide [475]. 5-Amino-4-pyrazole-carboxamides react with carbon disulfide in pyridine under reflux with loss of H_2S to give 6-thioxo-4,5,6,7-tetrahydro-4H-pyrazolo[3,4-d]-pyrimidin-4-ones in high yield [473].

Treatment of α-amino carboxylic acids **147** with carbon disulfide leads to 2-thioxo-5-thiazolidinones **140** via the intermediate dithiocarbamic acid **148**. Ring closure of the latter can be achieved occasionally by refluxing in HCl, but usually requires the addition of a dehydrating agent such as phosphorus tribromide or acetic anhydride [464, 469, 476–478].

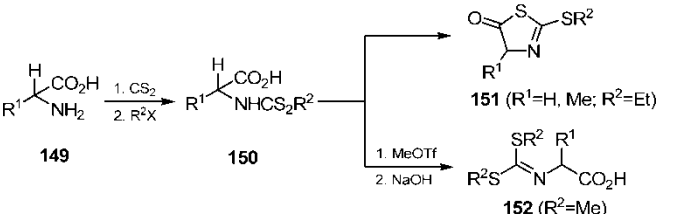


145 **146**

(X=S, NMe; R= SH, NHMe, SMe, CH_2Ph)

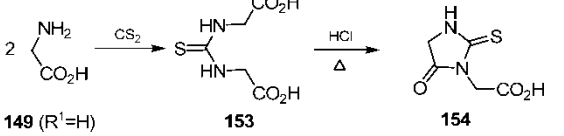


147 **148** **140**

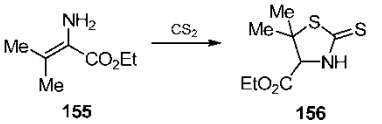


149 **150** **151** (R^1=H, Me; R^2=Et)

152 (R^2=Me)



149 (R^1=H) **153** **154**



155 **156**

Equimolar quantities of glycine or alanine **149** and carbon disulfide react in the presence of an alkylating agent to give S-alkyl dithiocarbamates **150** which can be cyclized to yield thiazolin-5-ones **151** [479, 480].

α-Amino [113, 481] and *ω*-amino carboxylic acids [482] react with carbon disulfide in the presence of triethylamine to afford dithiocarbamate intermediates which are easily alkylated *in situ* to yield **150**. Further alkylation with methyl triflate leads to the bis(methylthio)imines **152** [483].

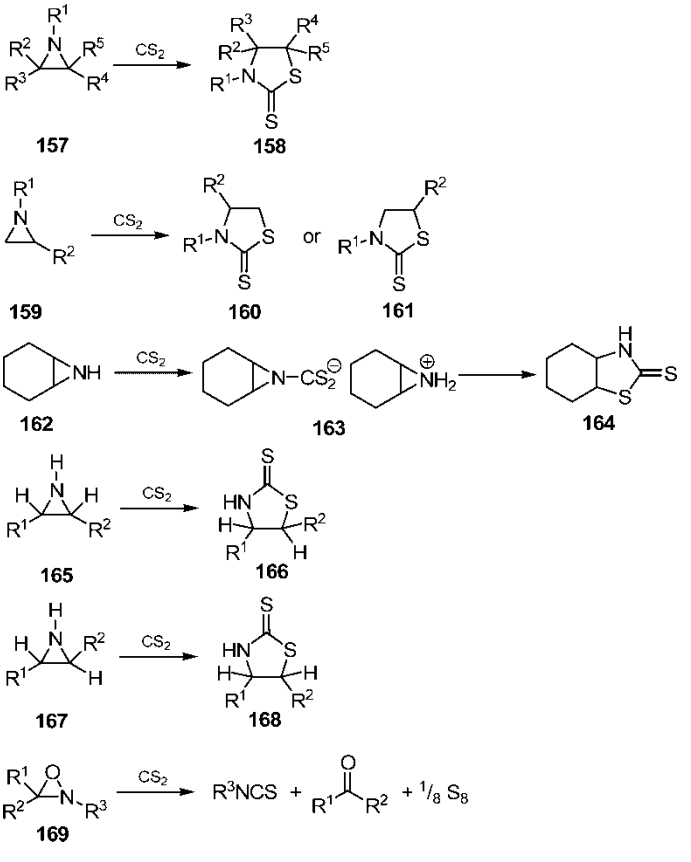
When 2 moles of glycine **149** (R^1=H) are treated with 1 mole of carbon disulfide, thiourea **153** is obtained which affords 2-thio-3-hydantoinacetic acid **154** on cyclization [484, 485].

Esters of α-amino acids react with carbon disulfide to yield 2-thioxo-5-thiazolidinones **140** [486]. The reaction of α,β-unsaturated amines **155** with carbon disulfide leads to thiazolidine-2-thiones **156** [487].

3. Reactions with three-membered ring nitrogen heterocycles

3.1 *Reactions with aziridines and oxaziridines*

The reaction, first investigated by Gabriel [448, 488, 489], of aziridines **157** (R^1=H) with carbon disulfide affords thiazolidine-2-thiones **158**. N-substituted aziridines **157** (R^1=alkyl) react analogously [490–496].



157 **158**

159 **160** **161**

162 **163** **164**

165 **166**

167 **168**

169

The reaction product depends on the substituents in the ring. Both 2-alkylaziridines **159** (R^1=H, R^2=alkyl) and their N-substituted analogues (R^1=alkyl, aryl or tosyl) give 4-substituted derivatives **160** corresponding to attack at the less hindered side [491–493, 497]. By contrast **159** (R^1=H, R^2=aryl) affords 5-aryl derivatives **161** indicating that ring opening proceeds at the 2-position in the aziridine [494]. In the presence of catalytic amounts of an organoantimony(V) halide the cycloaddition of aziridine **159** (R^1=Ph; R^2=Et) with carbon disulfide selectively gave ring-expanded cycloadduct **161** [498].

When N-arylaziridines **159** (R^1=aryl, R^2=H) are heated with carbon disulfide in a sealed tube in the presence of tetraethylammonium bromide, thiazolidines **160** [R^1=aryl, R^2=H (8–15%)] and a dithiocarbonate polymer (60–80%) are obtained [499].

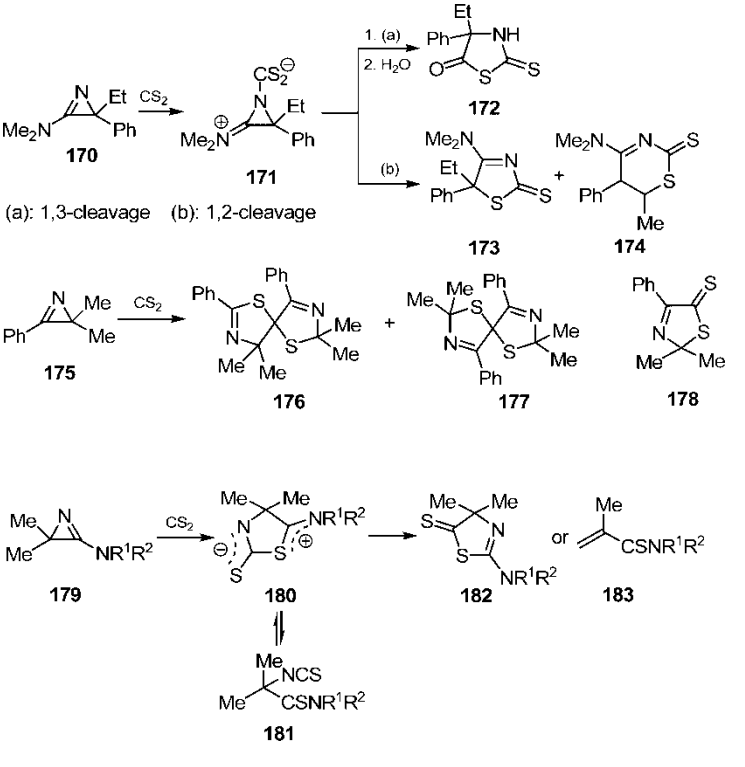
Treatment of the fused aziridine **162** with carbon disulfide yields the salt **163** which can be transformed to **164** which has *trans*-stereochemistry at the ring junction [290, 447]. Intermediate **163** can be isolated and thermolyzed to afford **164** along with **162**.

Reactions of both *cis*- and *trans*-2,3-dialkyl-aziridines with carbon disulfide have been investigated and it has been found that the geometries of the resulting thiazolidines are largely opposite to those of the original aziridines [495, 496]. Thus, reaction of the *cis*-isomer **165** proceeds stereospecifically to give the *trans*-thiazolidine **166**. Reaction of the *trans*-isomer **167** proceeds stereoselectively to yield the cis-thiazolidine **168**.

Carbon disulfide reacts with oxaziridines **169** (R^3=alkyl or cycloalkyl) to afford isothiocyanates in quantitative yield [500].

3.2 *Reactions with azirines*

Treatment of the 3-dimethylamino-2H-azirine **170** with carbon disulfide affords the dipolar adduct **171** which is capable of both 1,3- and 1,2-cleavage to afford **172** or **173** and **174** respectively [501].



Photolysis of 2H-azirine **175** in the presence of carbon disulfide gave 2:1 adducts **176** and **177**. The intermediacy of thiazolinethione **178** was postulated [502].

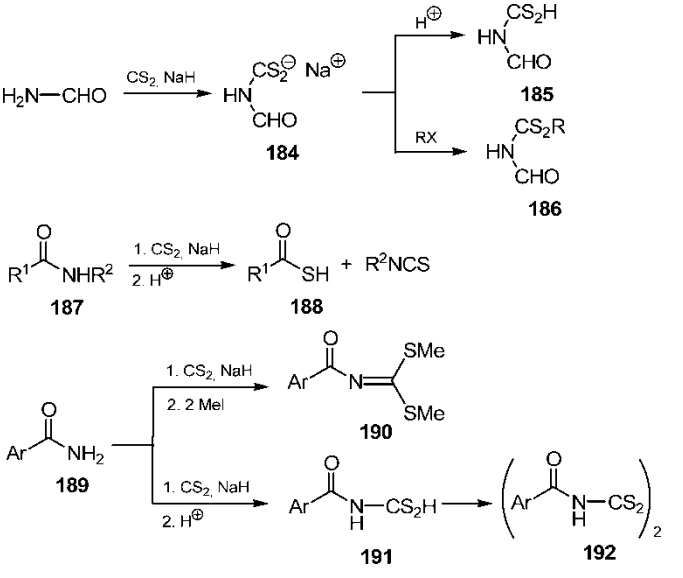
3-Dialkylamino-2H-azirines **179** react with carbon disulfide to afford 1:1 adducts which appear to exist as dipoles **180** in the solid state and as isothiocyanates **181** when molten [503, 504]. Thermolysis of the latter (R^1=R^2=Me or NR^1R^2=morpholino) yields the thiazoline **182**. Similar treatment of **180** (R^1=R^2=Et) produces the thioamide **183**.

4. Reactions with amides and derivatives

4.1 *Reactions with amides, imides and thioamides*

Treatment of formamide with carbon disulfide and NaH yields the dithiocarbamate salt **184** [505] which can be converted into corresponding acid **185** [505, 506] and esters **186** (R=Me or Et) [507].

Amide bonds in primary and secondary amides **187** (R^2=H or alkyl) can also be cleaved by carbon disulfide and NaH to yield, after acidification, thioacids **188** and isothiocyanates [508].

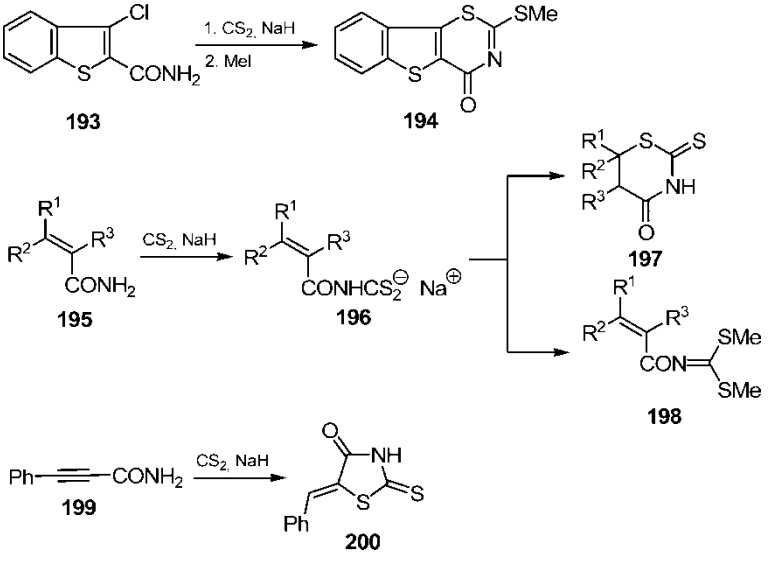


Aromatic amides **189** are converted into dimethyldithioimidocarbonates **190** on reaction with carbon disulfide, NaH and iodomethane (or dimethyl sulfate) [183, 509–512]. In the absence of iodomethane, acidification of the reaction mixture can, in some instances, yield free acids **191**, which undergo facile oxidative coupling in solution to give disulfides **192**.

Aromatic amides **193** containing *ortho*-halogen atoms can be converted into fused 1,3-thiazinones **194** on treatment with carbon disulfide and NaH followed by methylation [513].

Unsaturated amides **195** react with carbon disulfide/NaH to afford salts **196** which can be cyclized to tetrahydro-2-thioxo-1,3-thiazin-4-ones **197**, whilst dithioimidocarbonates **198** are obtained after methylation [514]. Cinnamide **195** (R^1=Ph, R^2=R^3=H) yields **198** (R^1=Ph, R^2=R^3=H) under similar reaction conditions [515]. 3-Phenyl-propynamide **199** affords the

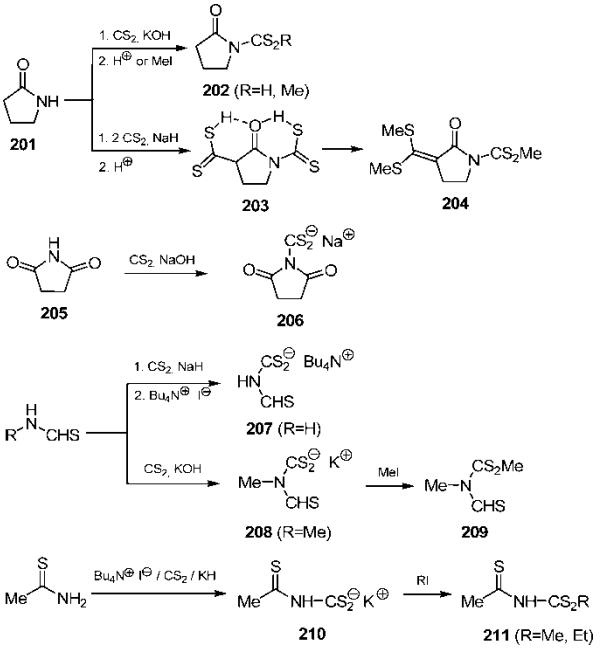
thioxothiazolidinone **200** [516].



Reactions of 2-pyrrolidone, 2-piperidone, ε-caprolactam, imidazolidin-2-one, imidazolidine-2,4-dione or 2,5-dioxopiperazine with carbon disulfide have been reported [38, 517]. 2-Pyrrolidone **201** reacts with carbon disulfide to give after acidification, the stable free acid **202** (R=H) which yields ester on treatment with iodomethane. By contrast, treatment of **201** with carbon disulfide and NaH and following acidification yield the 1,3-bisdithiocarboxylic acid **203**. Methylation produces the methyl 3-[bis(methylthio)methylene]-2-pyrrolidone-1-carbodithioate **204**.

Treatment of succinimide (or phthalimide) **205** with carbon disulfide/NaOH gives the salt **206** [518].

Thioformamide affords the dithiocarbamate salt **207** after reaction with carbon disulfide and NaH, followed by the addition of tetrabutylammonium iodide [519].



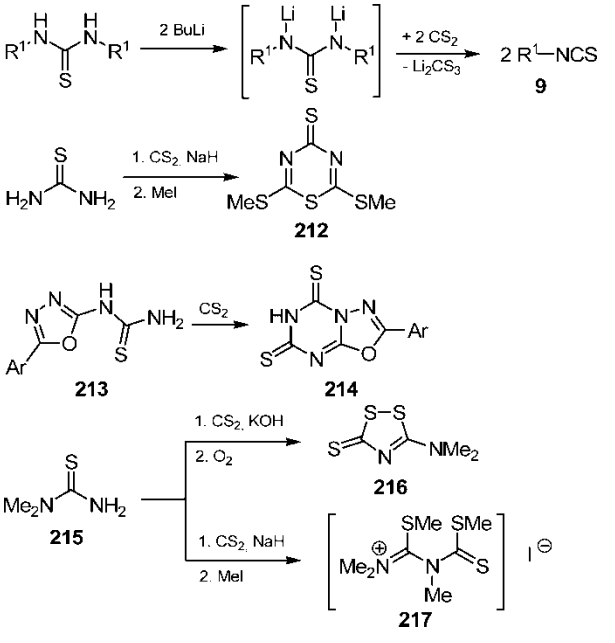
The S-methyl ester **209** of N-methyl N-thioformyl dithiocarbamic acid was prepared by reaction of potassium salt **208** with methyl iodide [520] .

Thioacetamide reacts with carbon disulfide in the presence of KH to form, via the tetra-Bu ammonium salt, N-thioacetyl dithiocarbamate **210**. The Me and Et esters **211** were obtained by esterifying with MeI or EtI. Attempts to synthesize N-thioacetyl dithiocarbamic acid were not successful [521].

4.2 *Reactions with ureas and thioureas*

The carbon-nitrogen bond in N,N′-diaryl- and dialkyl-thioureas or -ureas is easily cleaved by the metallation using butyllithium, followed by the addition of an excess of carbon disulfide under reflux, to give about two moles of the corresponding isothiocyanates **9** per mol of the thioureas or ureas used [522].

Treatment of thiourea with NaH, carbon disulfide and methyl iodide resulted in the formation of 1,3,5-thiadiazine-4-thione **212** [523].



Cyclocondensation of N1-(5-aryl-1,3,4-oxadiazol-2-yl)-thioureas **213** with carbon disulfide/KOH yielded 2-aryl-5H-1,3,4-oxadiazolo[3,2-a][1,3,5]triazine-5,7(6H)-dithiones **214** [524].

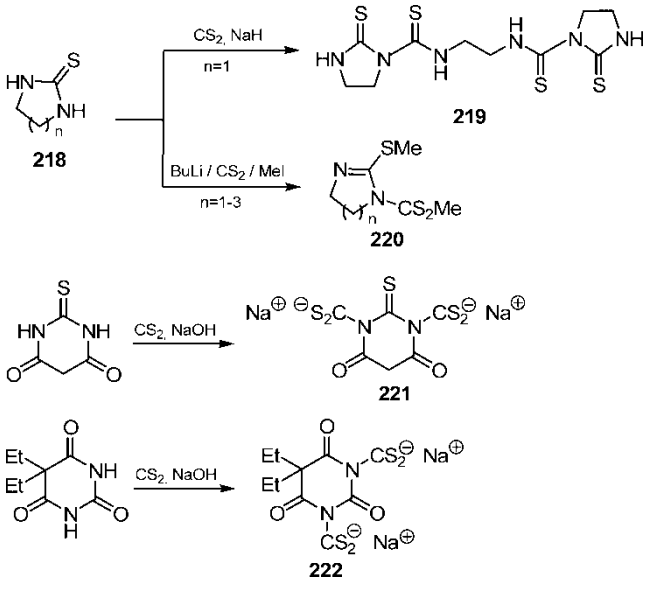
A number of heterocyclic systems are accessible from the reaction between carbon disulfide and N,N-substituted thioureas [525, 526]. Thus, treatment of **215** with carbon disulfide and KOH followed by oxidation affords the 1,2,4-dithiazole-3-thione **216**.

The immonium salt **217** result when **215** is treated with carbon disulfide, NaH and iodomethane [527, 528].

The cyclic thioureas **218** (n=1) afford **219** when reacted with carbon disulfide, and NaH [529, 530]. Treatment of **218** (n=1–3) with BuLi, CS_2, and MeI leads to isothioureas **220** [531].

Disodium 2-thio-4,6-pyrimidinedione-1, 3-bis-dithiocarboxylate **221** and disodium 5,5-diethyl-2,4,6-pyrimidinetrione-1, 3-bis-dithiocarboxylate **222** were synthesized by reaction

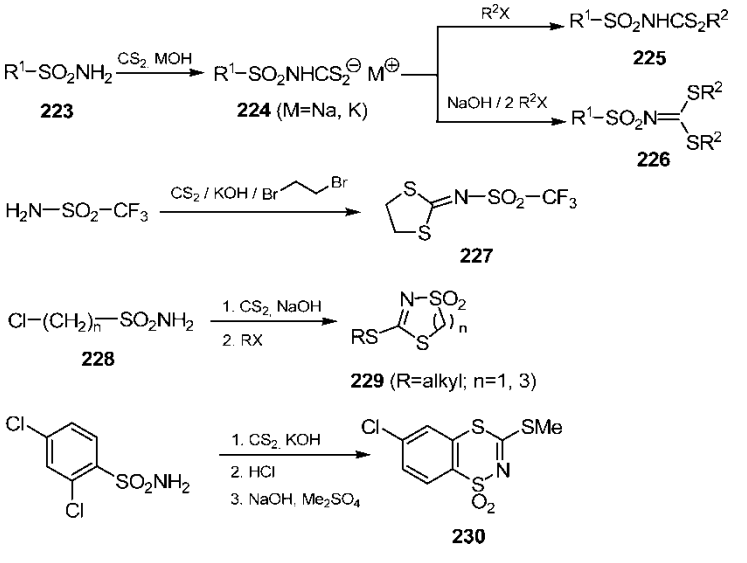
of barbituric acid derivatives with carbon disulfide [532].



4.3 *Reactions with sulfonamides*

Sulfonamides **223** react with carbon disulfide and NaOH, KOH, NaOMe or NaH to afford salts **224** which give esters **225** or dithioimidocarbonates **226** after alkylation [533–547].

N-(Trifluoromethylsulfonyl)dithioimidocarbonate **227** was prepared by condensation of carbon disulfide and trifluoromethylsulfonamide in the presence of potassium hydroxide with subsequent S-alkylation [548].

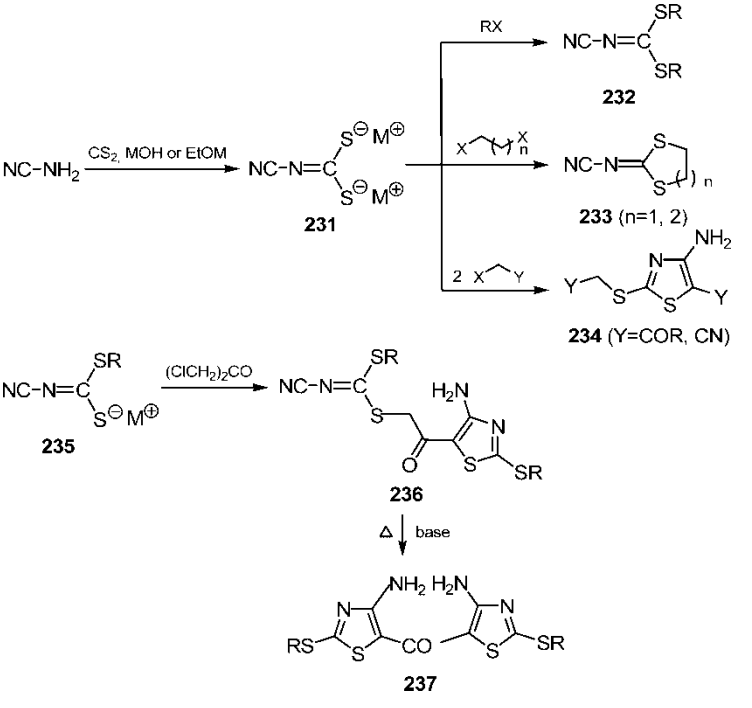


Chloroalkanesulfonamides **228** are converted into the 1,4,2-dithiazole-1,1-dioxide **229** (n=1) and 6,7-dihydro-5H-1,4,2-dithiazepine-1,1-dioxide **229** (n=3) by carbon disulfide and excess NaOH followed by alkylation [549, 550].

1,4,2-Benzodithiazine 1,1-dioxides of type **230** were obtained by reacting 2-chlorobenzenesulfonamides with carbon disulfide in the presence of potassium hydroxide [551].

4.4 *Reactions with cyanamide and cyanoguanidine*

Cyanamide reacts with carbon disulfide in the presence of metal hydroxides or alcoholates to give cyanoimidodithiocarbonate salts **231**. Various salts of this type have been described [34, 552–557]. Cyanoimidodithiocarbonates also result when calcium cyanamide is treated with carbon disulfide in the presence of a soluble metal carbonate [558–560].



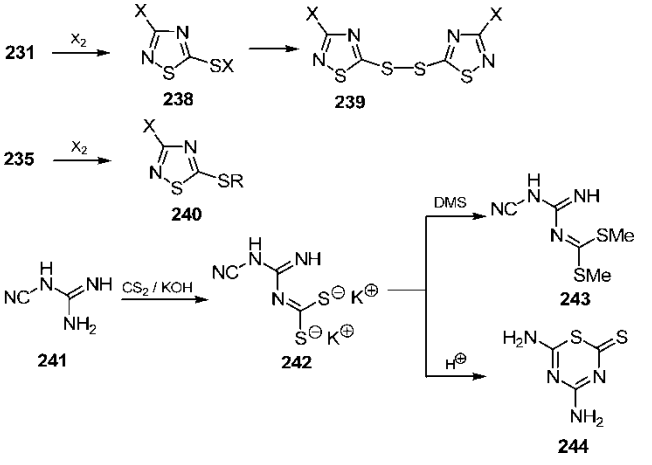
Salts **231** can be converted into cyanoimidodithiocarbonic acid esters **232** on treatment with alkylating agents [553, 561–570]. One-pot techniques using carbon disulfide, cyanamide and DMS in the presence of phase transfer catalyst have been reported [571, 572]. Alkylation with both 1,2- and 1,3-dihaloalkanes produces cyclic esters **233** (n = 1, 2) [554, 566, 573].

Cyanoimidodithiocarbonate salts **231** react with α-haloketones and haloacetonitriles to yield 4-aminothiazoles **234** (X=Cl, Br; Y=COR, CN) [558, 562, 574, 575].

Reaction of **235** (prepared by alkylation of **231** with one equivalent of haloalkane) with 1,3-dichloroacetone affords the thiazole **236** which can be cyclized under basic conditions to afford the ketone **237** [576].

Treatment of **231** and **235** with halogens or halogenating agents yields the 1,2,4-thiadiazoles **238** and **239**, and **240**, respectively [559, 564].

Cyanoguanidine **241** reacts with carbon disulfide to give the salt **242** which affords **243** on treatment with DMS [577–580]. Substituted cyanoguanidines react analogously [581].

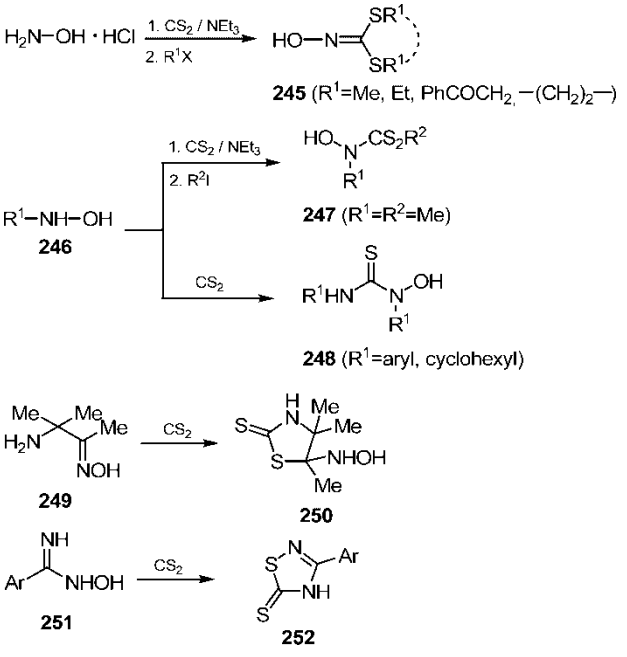


Cyclization of **242** with acidic reagents leads to the formation of thiadiazinethione **244** [578, 582, cf. 583].

5. Reactions with hydroxylamines and oximes

Hydroxylamine hydrochloride reacts with carbon disulfide in the presence of triethylamine to afford hydroxyimidodithiocarbonates **245** after alkylation [584–586].

The product from reactions involving N-alkyl analogues **246** depends on the reaction conditions [584, 587, 588]. In basic solution **246** reacts with carbon disulfide and iodomethane to yield dithiocarbamate **247**, whilst in DMF or DMSO solution, in the absence of base, cyclohexyl and aromatic hydroxylamines **246** yield N-hydroxythioureas **248**.

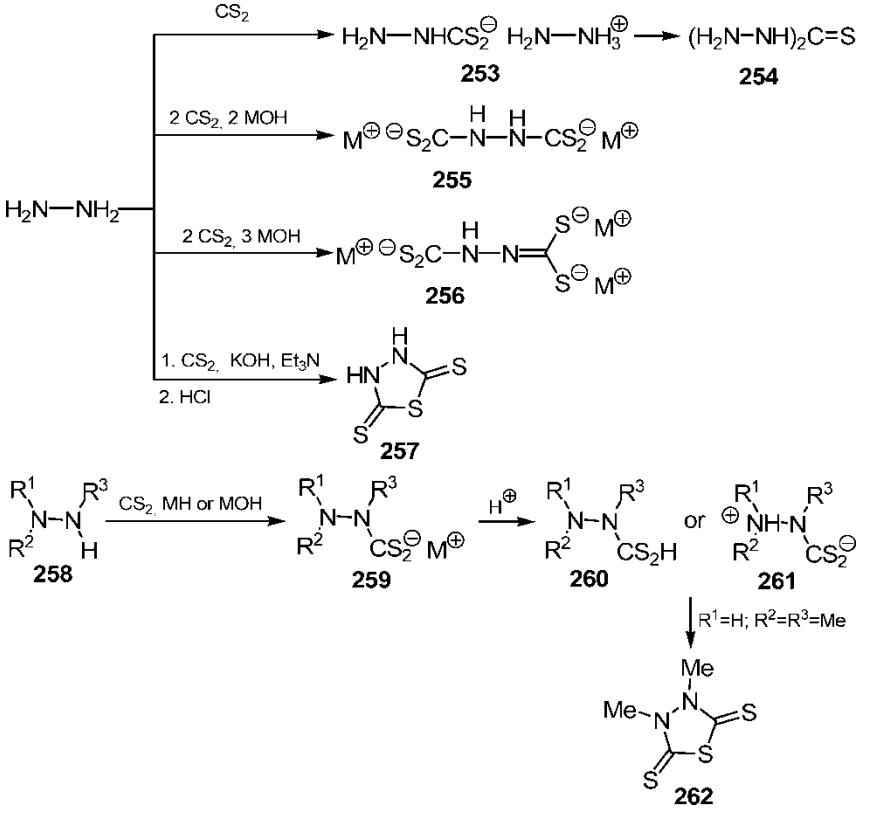


Amino substituted oxime **249** [309] and amidoximes **251** [589–591] react with carbon disulfide to give 5-(hydroxyamino)-thiazolidine-2-thione **250** and 1,2,4-thiadiazoline-5-thiones **252**, respectively.

6. Reactions with hydrazines, hydrazides and hydrazones

Since hydrazine possesses two primary amino groups, reaction with carbon disulfide can take place at one or both sites. In the absence of base, hydrazine reacts with carbon disulfide, to afford hydrazinium dithiocarbazate **253** [34] which decomposes in aqueous solution [592, 593] or on heating [594] to produce the thiocarbonohydrazide **254**. If however, the reaction is conducted in aqueous NaOH the reaction products are **255** or **256** depending on the concentration of the alkali [595–597]. 1,3,4-Thiadiazolidine-2,5-dithione **257** was synthesized from hydrazine hydrate, carbon disulfide, and KOH with triethylamine as a catalyst [275, 598].

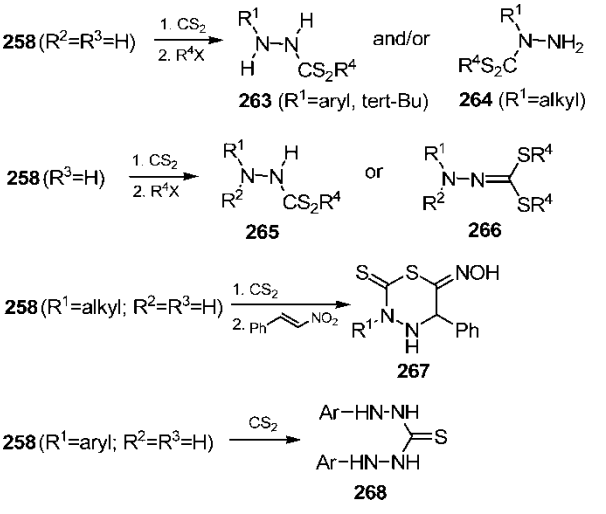
Dithiocarbazate salts **259** are produced on treatment of substituted hydrazines **258** with carbon disulfide, at low temperature, in the presence of base [34, 599]. The choice of base used depends on the basicity of the hydrazine. Reactions involving relatively basic hydrazines can be carried out in methanol using either NaOH or KOH, while with weakly basic hydrazines reactions are best conducted in DMSO using NaH [600].



Acidification of solutions of dithiocarbazates **259** affords free dithiocarbazic acids **260**. Thus, **260** (R^1=R^2=R^3=H) precipitates when its potassium salt is treated with dilute HCl at 0 °C [601]. Dithiocarbazic acids may adopt one of two structures **260** or **261**. In general, the dipolar structure **261** is favoured although **260** (R^1=R^2=Ph; R^3=H

or R^1=R^2=R^3=Me) is an exception [602, 603]. Dithiocarbazic acids are unstable and **261** (R^1=R^2=R^3=H) decomposes readily (even at −40 °C). **261** (R^1=H; R^2=R^3=Me) cannot be obtained pure, it evolves hydrogen sulfide to give 1,3,4-thiazolidine-2,5-dithione **262**.

Dithiocarbazate esters may be obtained by the alkylation of dithiocarbazate salts [600, 604–611]. Hydrazines **258** (R^1=aryl, tert-Bu; R^2=R^3=H) react with carbon disulfide to give, after alkylation, **263** whilst **258** (R^1=primary or secondary alkyl; R^2=R^3=H) yield esters **264** [604]. Benzylhydrazine produces both types [607]. An efficient method for the synthesis of dithiocarbazates **263** is the reaction of hydrazines with carbon disulfide and alkyl halides in the presence of cesium carbonate and tetrabutylammonium iodide at room temperature [612]. Hydrazinolysis of dithiocarbazate esters yields thiocarbohydrazides [613–615].



Reaction of N,N-disubstituted hydrazines **258** (R^3=H) or hydrazones with carbon disulfide followed by alkylation affords dithiocarbazate esters **265** or S,S-dialkyl esters **266** [612, 616–621].

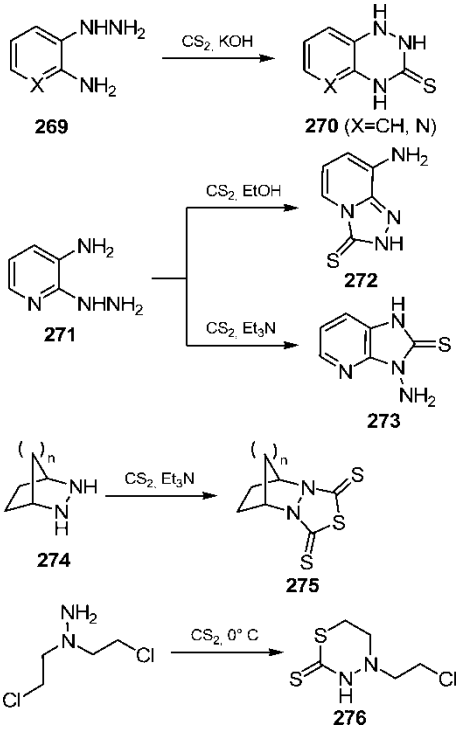
Aliphatic hydrazines **258** (R^1=alkyl; R^2=R^3=H) react with carbon disulfide and nitrostyrene to yield the 2-thioxo-tetrahydro-1,3,4-thiadiazin-6-one oximes **267** [622].

Carbon disulfide reacts with excess aryl hydrazines **258** (R^1=aryl) to give 1,5-diaryl-thiocarbohydrazides **268** [623, 624].

Aryl hydrazine **269** (X=CH) possessing *ortho*-amino group is transformed into 1,4-dihydro-1,2,4-benzotriazin-3(2H)-thione **270** (X=CH) [625]. 2-Amino-3-hydrazinopyridine **269** (X=N) reacts similarly to yield **270** (X=N) [626]. In contrast **271** affords the triazolo[4,3-a]pyridine-3(2H)-thione **272** when treated with carbon disulfide in ethanol solution and 1,3-dihydro-imidazo[4,5-b]pyridine-2-thione **273** when the reaction is conducted in the presence of triethylamine [627].

Several other nitrogen heterocycles containing 2-hydrazino groups react with carbon disulfide to afford 1,2,4-triazoles [275]. Examples which have been described include pyridine [628], quinoline [629], benzothiazole [629, 630], benzoxazole [629], benzimidazole [629],

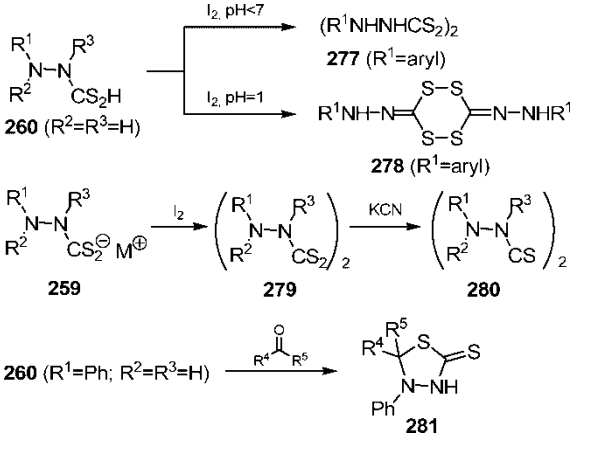
1,3,4-thiadiazole [631], pyridazine [632], and pyrimidine [629, 633, 634].

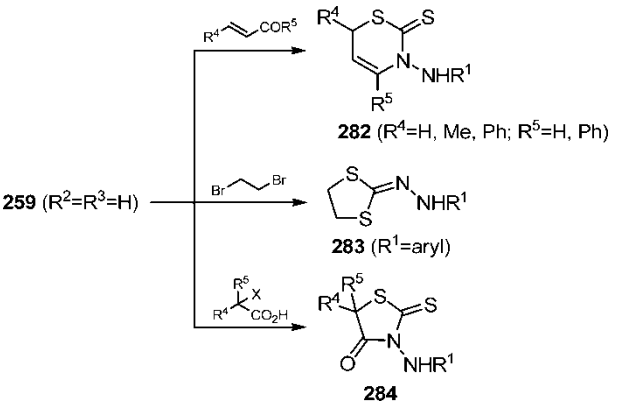


Fused 1,3,4-thiadiazolidine-2,5-dithiones **275** are prepared by treatment of cyclic hydrazines **274** with carbon disulfide [635] and N,N-bis(β-chloro-ethyl)hydrazine reacts to give the tetrahydro-1,3,4-thiadiazine-2-thione **276** [636].

Primary dithiocarbazic acids **260** (R^2=R^3=H) can be oxidized with hydrogen peroxide or iodine to yield either disulfides **277** or bis(arylhydrazones) of s-tetrathiane-3,6-dione **278** depending on the pH of the reaction mixture [637]. Oxidation of dithiocarbazate salts **259** produces disulfides **279** which can be partially desulfurized with KCN to give **280** [603].

3-Phenyldithiocarbazic acid **260** (R^1=Ph; R^2=R^3=H) reacts with aldehydes or ketones to produce 1,3,4-thiadiazolidine-2-thiones **281** [638–640].

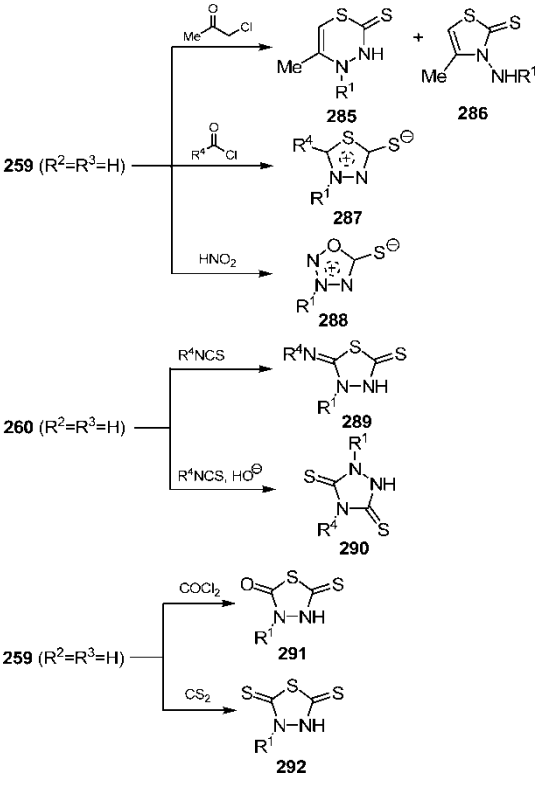




Reaction of dithiocarbazate salts **259** (R^2=R^3=H) with α,β-unsaturated aldehydes or ketones affords 3,6-dihydro-2H-1,3-thiazine-2-thiones **282** [641]. Reaction of **259** with reagents containing two reactive functional groups also leads to the formation of heterocycles. For example, **259** (R^1=aryl) reacts with 1,2-dibromoethane to yield hydrazones of 1,3-dithiolan-2-ones **283** [642]. Reaction with α-haloacids gives 2-thioxo-4-thiazolidinones **284** [643, 644].

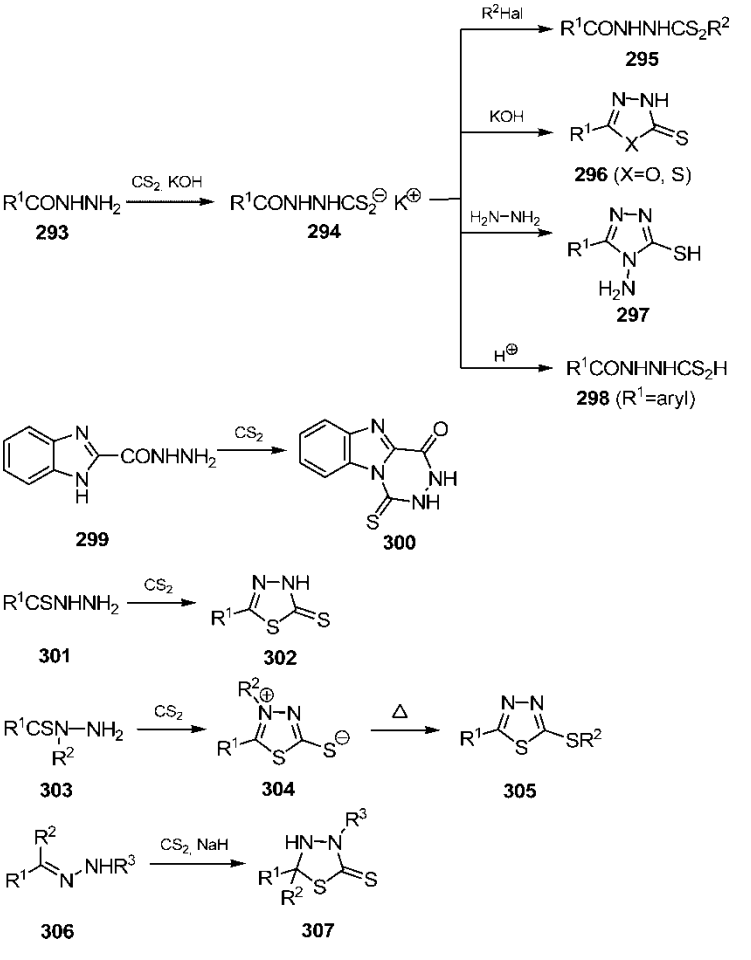
Ammonium dithiocarbazate **259** (R^2=R^3=H; M=ammonium) reacts with chloroacetone to yield 3,4-dihydro-2H-1,3,4-thiadiazine-2-thione **285** and 2(3H)-thiazolethione **286** [645, 646]. The major product depends on the conditions [645, 647–650]. Conversion of **285** to **286** can be accomplished by refluxing the former in alcoholic HC1.

Reaction of **259** (R^2=R^3=H) with acid chlorides or ethyl formamidate hydrochloride [640, 651–654], or nitrous acid [655] affords the mesoionic compounds **287** and **288,** respectively.



When acids **260** (R^2=R^3=H) are treated with isothiocyanates, 1,3,4-thiadiazolidine-2-thiones **289** or 1,2,4-triazolidine-3,5-dithiones **290** are obtained depending on the pH of the reaction mixture [656]. Reaction of **259** with phosgene [657] or carbon disulfide [275] produces 1,3,4-thiadiazolidines **291** and **292**. S-substituted derivatives of **292** were prepared from the reaction of monoalkyl hydrazines with carbon disulfide and organic halides in the presence of anhydrous potassium phosphate [658].

Hydrazides **293** react with carbon disulfide to afford dithiocarbazates **294** [659–664] which can be alkylated to give esters **295** [665]. Cyclization of **294** in basic solution yields 1,3,4-oxadiazole-2(3H)-thiones **296** (X=O) [666–669] or 1,3,4-thiadiazole-2(3H)-thiones **296** (X=S) [670, 671]. Treatment of **294** with hydrazine leads to 4-amino-1,2,4-triazole-3-thiols **297** [672]. The 3-aroyldithiocarbazates decompose near the equivalent point where an appreciable amount of the dithiocarbazic acid **298** is present [673, 674].



2-Benzimidazolecarboxylic acid hydrazide **299** affords the tricycle **300** [675]. Hydrazides of thiocarboxylic acids **301** are converted into 1,3,4-thiadiazole-2(3H)-thiones **302** [666, 676, 677].

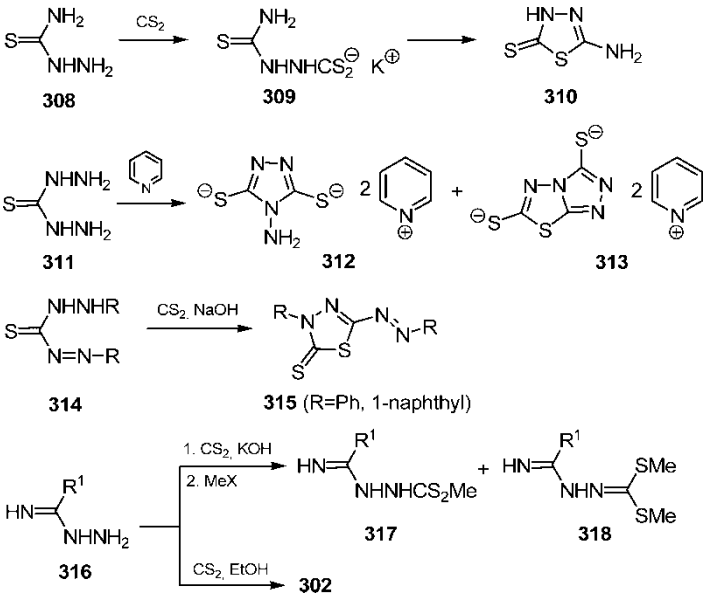
Similar treatment of the N-substituted hydrazides **303** produces mesoionic derivatives **304** which give 1,3,4-thiadiazoles **305** on heating [678].

Hydrazones **306** react with carbon disulfide to yield 1,3,4-thiadiazolidine-2-thiones **307** [679, 680].

7. Reactions with thiosemicarbazide, amidrazones and related compounds

Thiosemicarbazide **308** reacts with carbon disulfide and KOH in DMF [681] to afford the salt **309** which cyclizes on warming to give the 1,3,4-thiadiazoline-2-thione **310** [682–685]. By contrast, reaction of **308** with carbon disulfide and NaOH followed by acidification produces **257** [686]. Substituted thiosemicarbazides react analogously [682, 683, 687].

Reaction of thiocarbohydrazide **311** with carbon disulfide in pyridine affords the salts **312** and **313** [683].

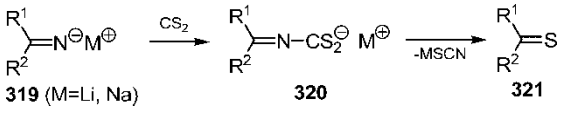


Heterocyclization of dithizone or di-naphthylthiocarbazone **314** with carbon disulfide leads to 3,5-substituted-1,3,4-thiadiazole-2(3H)-thiones **315** [688].

Addition product of carbon disulfide and amidrazone **316** can be alkylated to yield monoester **317** and/or [bis(methylthio)methylene]hydrazide **318**, whereas treatment of amidrazones with carbon disulfide in ethanol gives 5-substituted 1,3,4-thiadiazole-2(3H)-thiones **302** [689, 690].

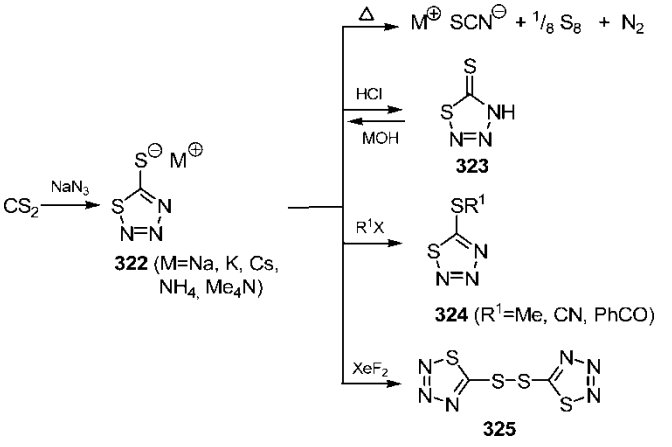
8. Reactions with imines

Imine salts **319** (M=Na or Li) react with carbon disulfide to afford intermediates **320** which lose thiocyanate ion to produce thioketones **321**. The method gives good results with both R^1=alkyl, R^2=alkyl or aryl and R^1R^2=cycloalkyl [691–694], but poor yields of aromatic thioketones (R^1=R^2=aryl) are obtained [695–697].



9. Reactions with azides

The 1,3-dipolar cycloaddition reaction between carbon disulfide and the azide anion yields the salt **322** [698, 699]. Addition of cold concentrated HCl affords 1,2,3,4-thiatriazole-5-thione **323** which is both shock and heat sensitive in the solid state [700, 701]. Salt **322** reacts with both alkylating and acylating agents, but the reaction site ($S-R^1$ or $N-R^1$ connectivity) has been the subject of some controversy [702–710]. The structure problem has now been resolved using X-ray diffraction techniques and it is firmly established that products are **324** (R^1=Me, CN, PhCO) arising from attack at the exocyclic sulfur atom [699, 711]. Salt **322** is easily oxidized to yield disulfide **325** [698, 711, 712].



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