DIVINYL SULFIDE.

15.* CYCLOADDITION OF DIVINYL SULFIDE AND ITS 2-METHYL DERIVATIVES TO THIOUREA AND N-MONOALKYL- AND N-MONOARYLTHIOUREAS

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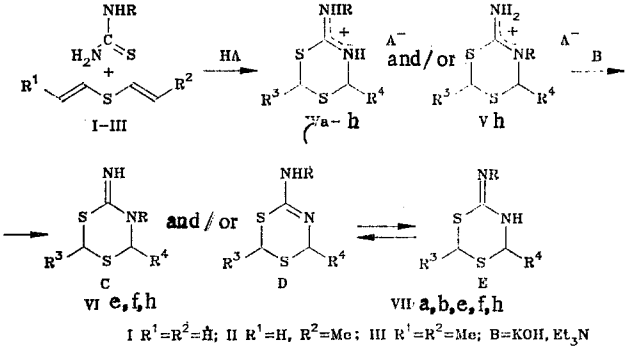
The heterocyclization of divinyl sulfide with N-monoalkyl- and N-monoarylthioureas and 2-vinyl propen-1-yl sulfide and di(propen-1-yl) sulfide with thiourea in the presence of equimolar amounts of inorganic acids leads to new nitrogen heterocycles 2H,6H-2,6-dialkyl-4-alkylamino- and 2H,6H-2,6-dialkyl-4-imino-5-N-phenyl-1,3,5-dithiazines in salt form. The action of bases on the diathiazine salts gives the corresponding 1,3,5-dithiazines. These heterocycles were found more sensitive to the action of nucleophiles causing ring opening than 1,3,5-dithiazines unsubstituted at the nitrogen atom.

In our previous work [2], we showed that divinyl sulfide (I) and thiourea undergo protophilic cyclization in the presence of acids to form 2H,6H-2,6-dimethyl-4-amino-1,3,5-dithiazinium salts. Divinyl sulfide derivatives with electron-withdrawing substituents such as di(2-phenylvinyl) sulfide do not undergo this reaction.

In light of the protophilic nature of this heterocyclization, we would have expected that the introduction of electron-donor substituents into the divinyl sulfide molecule would facilitate this process. We checked this proposal for the case of the reactions of vinyl propen-1-yl sulfide (II) and di(propen-1-yl) sulfide (III) with thiourea. Indeed, sulfides II and III under the same conditions used for divinyl sulfide (50-55°C, aqueous ethanol as solvent and acid catalysis) readily cyclize with thiourea to form the corresponding dithiazinium salts IV or Va-h in 60-90% yield.



I $R^1=R^2=H$; II $R^1=H$, $R^2=Me$; III $R^1=R^2=Me$; B=KOH, Et_3N

In the case of sulfide II, the formation of two structural isomers IVa and IVc or their mixtures may be formed, in which R^4 = Et may be either at $C_{(2)}$ or $C_{(6)}$ of the diathiazine ring (R = H).

Divinyl sulfide behaves analogously with N-monoalkyl and N-monoarylthioureas and two structural isomers with the Alk and Ph substituent either at the exocyclic (IVe-h) or endocyclic nitrogen (Ve-h, R^3 = R^4 = Me).

*For 14, see [1].

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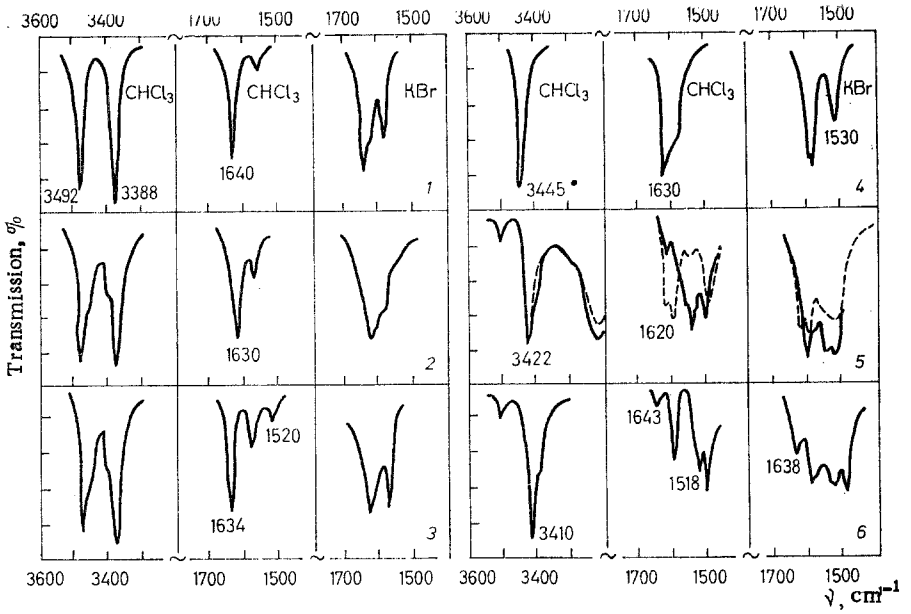


Fig. 1. IR spectra of 2H,6H-2,6-dialkyl-4-alkylamino- and 2H,6H-2,6-dialkyl-4-imino-5-N-phenyl-1,3,5-diathiazines: 1) 2H,6H-2,6-dimethyl-4-amino-1,3,5-dithiazine, 2) dithiazine VIIa, 3) dithiazine VIIb, 4) dithiazine VIIe, 5) dithiazine VIIf, 6) dithiazine VIh (see Table 1).

The physical constants and analytical data for the compounds synthesized are given in Table 1.

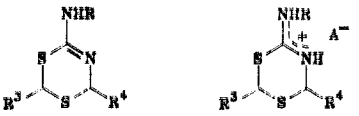
In addition ot the cyclization reaction, a side reaction involving the acid hydrolysis of the starting divinyl sulfides I-III is observed (sulfides I-III are thus taken in slight excess). Okuyama et al. [3] have reported that sulfides II and III are more stable toward acid hydrolysis than divinyl sulfide which, under the conditions of the reaction studied, partially undergoes hydrolytic cleavage to form H_2S, acetaldehyde and 2,4,6-trimethyl-1,3,5-trithiane [4].

Substituted divinyl sulfides II and III were used as isomer mixtures: 52% cis,cis and 48% trans,trans (II) and 40% cis,cis, 59% cis,trans, and 1% trans,trans (III) (determined by PMR spectroscopy). However, isomerically uniform dithiazinium salts IVa, b, e, f are obtained in all likelihood as a result of their cyclization with thiourea. The conclusion that these salts and their derived bases exist as single isomers is based on the lack of doubling of their PMR signals. Evidence for this conclusion is also found in the data of Day et al. [5] on the isomeric uniformity of dihydro-2,4,6-trimethyl-4H-1,3,5-dithiazone having only cis configuration as indicated by ^{13}C NMR spectroscopy and x-ray diffraction analysis.

The action of KOH or Et_3N on salts IVa, b, e, f, h gives 1,3,5-dithiazines VIIa, b, e, f and VIh (Table 1) which, similarly to these salts, are probably formed as structural isomers VId, f, h and VIIa, b, e, f, h. The latter may exist as two tautomeric forms (D and E).

While the neutralization of 2H,6H-2,6-dimethyl-4-amino-1,3,5-dithazinium nitrate [2] by 0.5 N aqeuous KOH proceeds smoothly with the formation of the free base, in the case of salts IVa-h, we must use weaker bases such as triethylamine in rigorously equimolar amounts since the dithiazines obtained are even more sensitive to the action of nucleophiles than 2H,6H-2,6-dimethyl-4-amino-1,3,5-dithiazine, which undergoes extensive decomposition by the action of excess amine [6]. We should note that products are formed in the reaction of dithiazinium salts IVa-h with triethylamine in water or nonabsolute solvents, whose IR spectra (for low concentrations in $CHCl_3$) show a strong band at 3505 cm^{-1} which does not disappear upon dilution of the solutions studied. The appearance of this band may be explained by strong intramolecular hydrogen bonding in trace hydroxyl-containing compounds formed as a result of the addition of water at the C=N bond of dithiazines VIIa-f and VIh with subsequent decomposition of the heterocycles. This band is lacking in the IR spectra of bases synthesized in dry solvents. Oligomerization of the dithiazines with ring opening is also possible in aqueous solvents. Thus, resins of various consistency are formed upon the preparation of dithiazine VIIf in nonabsolute acetone; the PMR spectra of these resins show broad peaks characteristic for oligomeric products at 1.18-1.55 (protons of three structurally inequivalent CH_3 groups),

TABLE 1. 2H,6H-2,6-Dialkyl-4-alkyl- and 2H,6H-2,6-4-Arylamino-1,3,5-dithazines and Their Salts



Compound*	T_{mp}, °C†	Found, %				Chemical formula	Calculated, %				Yield, %
		C	H(Cl)	N	S		C	H(Cl)	N	S	
IVa	155—157	—	(16,6)	13,1	30,6	$C_6H_{13}ClN_2S_2$	—	(16,7)	13,2	30,1	84
IVb	150—153	—	(15,0)	12,3	28,4	$C_7H_{15}ClN_2S_2$	—	(15,6)	12,4	28,3	80
IVc	Oil	30,9	5,8	16,7	25,9	$C_6H_{13}N_3O_3S_2$	30,1	5,5	17,6	26,7	62
IVd	98—102 (dec.)	33,1	6,1	16,5	25,5	$C_7H_{15}N_3O_3S_2$	33,2	5,9	16,6	25,3	68
IVe	Oil	—	(16,7)	12,6	29,5	$C_6H_{13}ClN_2S_2$	—	(16,7)	13,2	30,1	77
IVf	118—120	—	(14,8)	12,7	27,9	$C_7H_{15}ClN_2S_2$	—	(15,6)	12,4	28,3	94
IVg	Oil	—	(11,0)	8,9	20,5	$C_{13}H_{27}ClN_2S_2$	—	(11,4)	9,0	20,6	90
IVh	Powder	—	(13,6)	10,3	23,5	$C_{11}H_{15}ClN_2S_2$	—	(12,9)	10,2	23,3	90
VIIa	Oil	40,7	6,8	15,2	37,0	$C_6H_{12}N_2S_2$	40,9	6,9	15,9	36,4	68
VIIb	52—53	43,9	7,4	14,6	33,9	$C_7H_{14}N_2S_2$	44,2	7,4	14,7	33,7	94
VIIc	95—96	40,8	6,9	16,0	36,5	$C_6H_{12}N_2S_2$	40,9	6,9	15,9	36,4	66
VIId	Oil	43,5	7,5	14,7	33,3	$C_7H_{14}N_2S_2$	44,2	7,4	14,7	33,7	64
VIe	Oil	56,0	6,2	12,0	26,4	$C_{11}H_{14}N_2S_2$	55,4	5,9	11,8	26,9	96

*IVa-d, VIIa, b R = H, IVe, VIIe R = Me, IVf, VIIf R = Et, IVg R = octyl, IVh, VIh R = Ph, IVa, c, e-h, VIIa, e, f, VIh R^3 = Me, IVb, d, VIIb R^3 = Et, IVa-d, VIIa, b R^4 = Et, IVe-h VIIe, f, VIh R^4 = Me, IVa, b, e-h A = Cl, IVc, d A = NO_3.
†IVa was crystallized from aqueous ethyl acetate, IVb from ethanol, IVd, f from acetone, and VIIb, e from CCl_4.

3.49 ($N-CH_2CH_3$ methylene protons), and 4.50 ppm ($CHCH_3$ methine protons). On the other hand, the mass spectrum shows a molecular ion with m/z 191 corresponding to the mass of the monomer, which indicates the presence of monomer molecules in the oligomer or depolymerization of the oligomer upon electron impact. The IR spectrum of this sample also differs from that for the same dithiazine synthesized in absolute ether (Fig. 1).

In order to confirm the structures of dithiazines VIIa-f and VIh and study their tautomeric transformations, we investigated their IR spectra in chloroform, acetonitrile, and KBr pellets (Fig. 1). Conclusions regarding the structures of these compounds may be made by comparing the NH stretching and deformation bands and double bond stretching bands.

Considerable difficulties arise in the IR spectra of such compounds since the characteristic bands of amino and imino forms are found close to each other [7, 8]. Thus, we used the IR and UV spectra of 2H,6H-2,6-dimethyl-4-amino-1,3,5-dithiazine (VIII) and its acyl derivatives [8, 9], which are similar in structure and method of synthesis with the compounds studied. The introduction of an ethyl group at $C_{(2)}$ or $C_{(6)}$ of the dithiazine ring (VIIa) (the position of the substituent was not unequivocally indicated by the PMR spectrum) leads to changes only in the NH_2 stretching band region. Figure 1 shows that the spectrum of VIIa has bands at 3388 and 3487 cm^{-1}, and the band at 3487 cm^{-1} is asymmetric while the band at 3388 cm^{-1} has a pronounced shoulder at 3410 cm^{-1}, which indicates the existence of an additional two NH groups, apparently due to imino form E since these bands for VIII with amino structure are symmetric (ν_s 3388 and ν_{as} 3492 cm^{-1}) [8]. Thus the bands for ν_s 3388 and ν_{as} 3487 cm^{-1} indicate the presence of amino form D. The band at ~3410 cm^{-1} may be assigned to stretching vibrations of the =NH group, while the absorption band of the secondary NH group (imino form E) probably overlaps the band at 3487 cm^{-1}.

The spectrum of dithiazine VIIb (R = H, R^3 = R^4 = Et) shows bands characteristic for imino form E (in $CHCl_3$ solution) even in the double bond region as indicated by the weak band at 1520 cm^{-1} due to stretching vibrations of the exocyclic C=N group of the imino form [9-11]. In general, however, the IR spectrum of this compound indicates that it exists in amino form D (Fig. 1). The predominance of amino form D for VIIb in CCl_4 is also indicated by its PMR spectrum, which shows a broad singlet for the NH_2 group at 5.08 ppm with integral intensity corresponding to two protons.

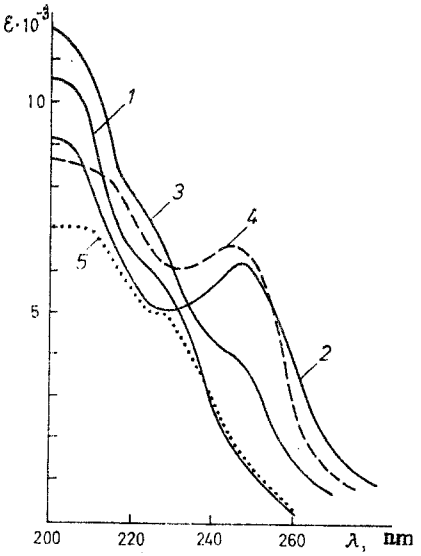


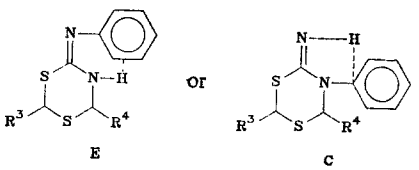
Fig. 2. UV spectra of 2H-6H-2,6-dialkyl-4-alkylamino-1,3,5-dithiazines: 1) 2H,6H-2,6-dialkyl-4-amino-1,3,5-dithiazine, 2) dithiazine VIIa, 3) dithiazine VIIb, 4) dithiazine VIIe, 5) dithiazine VIIf (See Table 1).

The IR spectrum of dithiazine VIIe (R = R^3 = R^4 = Me) in KBr pellets shows a band at 1530 cm^{-1} which may be assigned, as in the case of VIIb, to the stretching vibrations of the exocyclic C=N group of imino form E. In going from the crystal to solutions in chloroform and acetonitrile, changes are encountered in the spectra of VIIe: The band at 1530 cm^{-1} disappears and a band appears at 1630 cm^{-1} assigned to the ring C=N group [9, 10]. The only band in the high-frequency region at 3445 cm^{-1} may be due to vibrations of free NH in amino form D. These results permit us to exclude the structure with substituent R = Me at the endocyclic nitrogen atom VIe, since such a structure may exist only in imino form C and cannot take part in tautomeric transformations.

The PMR spectrum of the CCl_4 solution of VIIe has only one sharp signal for the methyl group protons at the nitrogen atom at 2.81 ppm. The single broad NH proton signal is at considerably higher field (3.88 ppm) relative to dithiazine VIIb which does not have an electron-donor alkyl substituent at the nitrogen atom. We should also note the significant chemical inequivalence of the methine protons (see Experimental section). All these findings indicate the existence of dithiazine VIIe in CCl_4 solution predominantly in amino form D [12].

The presence of a more bulky substituent (R = Et, VIIf) at the nitrogen atom complicates the IR spectrum of this compound at 1500-1600 cm^{-1}. One broad band is found in the spectrum for the compound in a KBr pellet; this band has several maxima. In chloroform solution, the band at 1550 cm^{-1} decreases markedly in intensity while the band at 1627 cm^{-1} becomes more intense, which also indicates the presence of significant amounts of amino form D in this dithiazine. The broader band at 3222 cm^{-1} does not disappear with decreasing concentration from 0.02 to 0.002 mole/liter. This behavior is likely the results of strong intermolecular hydrogen bonding.

The IR spectrum of VIh with an electron-withdrawing substituent (R = C_6H_5) both neat (film upon evaporation of $CHCl_3$) and in $CHCl_3$ solution shows a strong band at 1518 cm^{-1} and a weak band at 1643 cm^{-1}. The predominance of the imino form is also indicated by the band at 3410 cm^{-1} (this band should be significantly higher for ArNHR [13]). The band at 3385 cm^{-1} may be assigned to an intramolecular hydrogen bond between the NH group and the benzene ring. This type of hydrogen bonding is possible in a structure with an exocyclic C=N bond.



E C

In light of the IR data and the protophilic nature of the cyclization as well as the more "acidic" properties of the hydrogen at a nitrogen atom adjacent to a phenyl ring than for NH_2 hydrogens in N-phenylthiourea, we may assume the formation of dithiazine VIh in imino form C.

These conclusions concerning the structure and tautomerism of dithiazines VIIa, b, e, f, h were supported by their UV spectra which show three bands (200-210, 210-230, and 240-260 nm, Fig. 2). In our previous work [9], we showed that the bands at 210-230 nm may be assigned to amino form D while the bands at 240-260 nm may be assigned to imino form E.

EXPERIMENTAL

The PMR spectra of 10-20% solutions of IVa, b, e, f and VIIa, b, e, f, h in CCl_4 and CD_3OD were taken at room temperature on a Tesla BS-497-C spectrometer at 100 MHz with TMS as the internal standard. The IR spectra were taken on a Specord 75-IR spectrometer in the range from 700 to 3700 cm^{-1} in KBr pellets, chloroform, and acetonitrile (c 0.2-0.002 mole/liter, J 0.4-2 cm). The UV spectra were taken in ethanol on a Specord-vis spectrophotometer.

A sample of di(propen-1-yl) sulfide was obtained according to our previous procedure [14]. Freshly distilled vinyl propen-1-yl sulfide (bp 38-40°C (53.3 hPa)) and di(propen-1-yl) sulfide (bp 45°C (20 hPa)) were used in the reactions.

Monosubstituted N-alkyl and N-phenylthioureas were obtained according to standard procedures [15]. N-Methylthiourea, mp 119-121.5°C (from ethanol), 119-120.5°C (lit. value). N-ethylthiourea, mp 103-106°C, 103-106°C (lit. value). N-octylthiourea, mp 84-85°C (from ethanol) (Found: C 57.5; H 10.7; N, 14.1; S, 16.9%. Calculated for $C_9H_{20}N_2S$: C 57.4; H 10.7; N 14.9; S, 17%), mp 114 [16]. N-Phenylthiourea, mp 152-153°C (from ethanol), 152.5-153°C (lit. value).

Reaction of Vinyl Propen-1-yl Sulfide and Di(propen-1-yl) Sulfide with Thiourea in the Presence of Acids (General Method). A sample of 8.3 g (110 mmoles) thiourea and 80 ml alcohol (ethanol or methanol) were added to a solution of 110 mmoles acid in 20 ml water and stirred for 0.5 h at 20°C. The temperature was raised to 55°C and 14.7 g (130 mmoles) di(propen-1-yl) sulfide was added. The mixture was stirred at 55°C for 6 h. Most of the solvent was removed in vacuum. The crystalline precipitate (7.6 g) was separated. More crystals were obtained upon letting the mother liquor stand. The reaction with HCl gave 12.2 g 2H,6H-2,6-diethyl-4-amino-1,3,5-dithiazinium chloride. PMR spectrum (CD_3OD): 1.15, overlap of two triplets (6H, $CHCH_2\underline{CH_3}$), 2.03, overlap of two multiplets (4H, $\underline{CH}\text{—}\underline{CH_2}CH_3$), 5.05 δ, ppm, q formed by the overlap of two triplets (2H, $\underline{CH}CH_2CH_3$).

The PMR spectra of the dithiazines studied and their salts feature overlap of signals in the corresponding spectral regions due to the structural inequivalence of the methine, methylene and methyl protons of the $XCHCH_3$ and $XCHC_2H_5$ groups (X = N, S). In comparison with the PMR spectra of the free bases, all the protons of the CH, CH_3, and C_2H_5 groups of the corresponding salts are shifted downfield. The endocyclic nitrogen atom is the protonation site in these heterocycles, which is in accord with our previous data [17] on the enhanced basicity of this nitrogen atom in 2H,6H-2,6-dimethyl-4-amino-1,3,5-dithiazine.

The reactions of vinyl propen-1-yl sulfide with thiourea and divinyl sulfide with N-alkyl- and N-phenylthiorueas were carried out by analogous procedures.

2H,6H-2,6-Diethyl-4-amino-1,3,5-dithiazine (VIIb). A sample of 1.83 g (18 mmoles) triethylamine in 10 ml ether was added with stirring to 4.1 g (18 mmoles) chloride IVb in 30 ml ether. Stirring was continued for an additional 4 h at room temperature. Then, the precipitate of $N(C_2H_5)_3 \cdot HCl$ was separated. Ether was removed in vacuum to give 3.2 g (94%) base VIIb. PMR spectrum (solution of VIIb in CCl_4), δ: 1.11 (m, 6H, $C_2\underline{CH_3}$), 1.87 (m, 4H, $\underline{CH_2}CH_3$), 4.48 (q, 2H, N $X\underline{CH}C_2H_5$, X = N and S), 5.08 ppm (br. s, 2H, NH_2).

2H,6H-2,6-Dimethyl-4-methylamino-1,3,5-dithiazine (VIIe). A sample of 56.4 ml 0.5 N aqueous KOH (28 mmoles) was added with stirring to 6 g (28 mmoles) chloride IVe in 30 ml water. Stirring was continued for 4 h at room temperature. The reaction mixture was extracted with four 50-ml portions of ether. The solvent was removed in vacuum to give 3.3 g (66%) base VIIe, which was twice crystallized from CCl_4 (Table 1). PMR spectrum (CCl_4), δ: 1.52 (d, 6H, $XCH\underline{CH_3}$, X = N and S), 2.82 (s, 3H, $NH\underline{CH_3}$), 3.88 (br. s, 1H, NH), 4.41 (q, 1H, $S\underline{CH}CH_3$), 4.66 ppm (q, 1H, $N\underline{CH}CH_3$), $^3J_{XCHCH_3}$ = 6.5 Hz.

2H,6H-2,6-Dimethyl-4-ethylamino-1,3,5-dithiazine (VIIf). A sample of 1.3 g (13 mmoles) triethylamine in 5 ml ether was added with stirring to 3 g (13 mmoles) chloride IVf in 30 ml dry ether, and stirring was continued for 4 h at 20°C. The $N(C_2H_5)_3 \cdot HCl$ precipitate was filtered off and washed with ether. The solvent was removed in vacuum to give 1.6 g (64%) base VIIf as a viscous oil.

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MASS-SPECTROMETRIC STUDY OF BENZOPYRIDOSILAAZEPINES AND -AZEPINONES

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The influence of various structural factors on the dissociative ionization of benzopyridosilaazepines and -azepinones has been investigated. It has been shown that the mass spectra can be used to identify isomeric benzopyridosilaazepinones with respect to the position of the amide fragment in the central heterocycle. The anomalously high intensity of the ion $[M - H]^+$ in the mass spectra of these compounds is attributed to fragmentation of the molecular ions from the open form.

The dissociative ionization of benzo[b,f]silepines [1] (nitrogen-free analogs of the substances investigated in the present article) has been reported earlier [1]. The mass-spectrometric characteristics of polycyclic compounds which contain the silaazepine fragment have so far not been studied. In the present work we have investigated fragmentation of the

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