X-ray crystallographic studies on cis N-(1, 1,2,2-tetrachloroethylthio) -4-cyclohexene-1,2-dicarboximide)

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ABSTRACT

In recent part it has been observed that some of these fungicides are loosing their effects and becoming resistant to them. Analogous compounds can be designed as substitute, if their structures are known. A rational approach to test these fungicides is to know the three dimensional structure of these compounds and macromolecular receptor sites as well as their molecular complex The structures of these compounds can be obtained by X-ray diffraction method in crystalline form and they will invariably be similar to their structure in solutions. The composition of cis N-(1, 1, 2, 2-tetrachloroethylthio)-4-cyclohexene-1, 2-dicarboximide) crystals are confirmed by comparing the infra-red spectra of the two components. The Unit cell parameters are a=10.5665(7)Å, b=6.6413(3)Å, c=19.3973(12)Å and Z=4. Thus the space group is determined to be $P2_1/c$ and crystal of monoclinic system.   © 2008 Trade Science Inc. - INDIA

KEYWORDS

INTRODUCTION

Captafol is a systemic fungicidal compound. Chemically it is cis N-(1, 1,2,2-tetrachloroethylthio)-4-cyclohexene-1,2-dicarboximide). Its molecular formula is $C_{10}H_9Cl_4NSO_2$ and molecular weight is 349.1. Although it is mainly recommended for foliar sprays, it has been used for seed dressing as well as soil application. It is available mostly as a 80 percent Weltable Powder. Crystallization was done by slow evaporation from a solution of methyl alcohol at 40°C temp. The crystals found were pale yellow in color and rectangular in shape. The density of the crystal is determined by floatation method at room temperature . The measured and calculated densities were $1.712 mg/m^3$ and $1.704 mg/m^3$ respectively. Chemical structure of cis N-(1, 1, 2,2-tetrachloroethylthio)-4-cyclohexene-1,2-dicarboximide) is given in figure 1.

The preliminary information about crystal is given in TABLE 1.
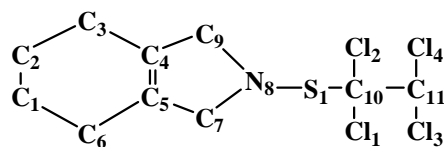
Data collection and structure solution

Figure 1: Numbering scheme cis-N-(1,1,2,2-tetrachloroethylthio)-4-cyclohexene-1,2-dicarboximide

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TABLE 1

Preliminary	Crystal deata
Chemical name	cis N-(1, 1 ,2,2-tetrachloroethylthio)-4-cyclohexene-1 ,2-dicarboximide)
Chemical formula	$C_{10} H_9 Cl_4 NSO_2$
Molecular weight	49.1
System	Monoclinic
Space group	$P2_1/c$
A	10.566(7)Å
B	6. 641(3)Å
C	19.397(12)Å
α	90°
β	91.79(10)°
γ	90(1)°
V	1360.84 Å^3
Dm	1.712mg/m^3
Dc	1.704mg/m^3
λ(Mokα)	71073Å
Z	4
Mode of data	CAD-4Enraf Nonious 4-circle automatic diffractometer
Structure solution	SHELXS-97
Structure refinemen t	SHELXL-97
Mode of data collection	ω-2θ
Theta range	1-73°
No. of reflections measured	8018
No. of unique reflections	3114
Temp. of crystal during Data collection	293°K
μ	0.260mm^{-1}
Lp correction	Applied
Absorption coefficient	Not applied

The three dimensional intensity data were collected on a computerized automatic 4-circled CAD-4 Enraf-Nonious Diffractometer using at the Deptt. of Biophysics AIIMS, New Delhi. Temperature of the crystal during data collection was 293°K. All the data were corrected for Lorentz and Polarization effect, but no absorption corrections were applied. Attenuation of the beam for measuring the strong reflections was accomplished by inserting calibrated pack varying from 3 to 10 as required Some standard reflections were measured for which h varies from -12 to 13, K varies from -8 to 8 and I varies from -25 to 21. The total numbers of unique reflections were 3114. Each intensity measurement involved in a scan over the reflection peak, a back ground measurement at each end of the scan range and a measurement of the peak height. The crystal is in monoclinic system and space group P2 The crystal structure was solved using SHELXS-97, programme.

TABLE 2 : Atomic Coordinates (×10^4) and equivalent isotropic displacement parameters (A^2×10^3) for Captafol. U(eq) is defined as one third of the trace of the orghogonalized Uij tensor

Atom	x	y	z	U (eq)
S(1)	3738 (1)	1230 (1)	1149 (1)	39 (1)
C1 (1)	2563 (1)	615 (1)	-146 (1)	60 (1)
C1 (2)	990 (1)	444 (1)	1051 (1)	59 (1)
C1 (3)	4109(1)	-3245 (1)	432 (1)	57 (1)
C1 (4)	1410 (1)	-3813 (1)	335 (1)	69 (1)
C (1)	867 (2)	-377 (5)	3135 (1)	55 (1)
C (2)	1519 (3)	-2053 (4)	3159 (1)	54 (1)
C (3)	2853 (2)	-2043 (4)	3425 (1)	50 (1)
C (4)	3573 (2)	-240 (3)	3137 (1)	35 (1)
C (5	2832 (2)	1745 (3)	3124 (1)	36 (1)
C (6)	1462 (2)	1550 (4)	3370 (1)	52 (1)
C (7)	2832 (2	2456 (3)	2382 (1)	34 (1)
N (8)	3411 (2)	939 (3)	1988 (1)	31 (1)
C (9)	3886 (2)	-639 (3)	2399 (1)	32 (1)
C (10)	2475 (2)	-207 (3)	720 (1)	36 (1)
C (11)	2651 (2)	-2500 (3)	770 (1)	40 (1)
C (12)	4433 (2)	-2069 (3)	2173 (1)	50 (1)
O (13)	2426 92)	4002 (2)	2149 (1)	51 (1)

TABEL 3 : Anisotropic displacement parameters (A^2×10^3) for Captafol. The anisotropic displacement factor exponent takes the form: $-2 pi^2 [h^2a^{*2} U11 + ... + 2h k a^* b^* U12)$

Atom	U11	U22	U33	U23	U13	U12
S (1)	37 (1)	44 (1)	35 (1)	1 (1)	5 (1)	-10 (1)
C1 (1)	93 (1)	56 (1)	32 (1)	6 (1)	-6 (1)	7 (1)
C1 (2)	31 (1)	79 (1)	68 (1)	-21 (1)	1 (1)	6 (1)
C1 (3)	56 (1)	58 (1)	58 (1)	-9 (1)	5 (1)	18 (1)
C1 (4)	68 (1)	64 (1)	75 (1)	-18 (1)	-7 (1)	- 23 (1)
C1 (1)	31 (1)	75 (2)	58 (2)	-2(1)	5 (1)	-14(1)
C (2)	50(1)	49(2)	62 (2)	-7(1)	14 (1)	-21(1)
C (3)	56 (2)	44(1)	49(1)	11(1)	9(1)	-2(1)
C(4)	28(1)	41(1	35(1)	0(1)	-5(1)	-2(1)
C(5)	37(1)	35(1)	36(1)	-8(1)	1(1)	-5(1)
C(6)	46 (1)	52 (2)	59 (2)	-9(1)	20 (1)	4 (1)
C (7)	32 (1)	31 (1)	40 (1)	-6 (1)	0 (1)	-2 (1)
N (8)	27 (1)	33 (1)	32 (1)	-2 (1)	-1 (1)	0 (1)
C (9)	23 (1)	37 (1)	37 (1)	0 (1)	-3 (1)	2 (1)
C (10)	33 (1)	44 (1)	30 (1)	0 (1)	0 (1)	3 (1)
C (11)	40 (1)	42 (1)	39 (1)	- 1 (1)	0 (1)	-3 (1)
O (12)	46 (1)	51 (1)	53 (1)	-4 (1)	2 (1)	22 (1)
O (13)	64 (1)	34 (1)	56 (1)	-1 (1)	-2 (1)	12 (1)

Refinement

From the SHELXS-97 programme for the crystal structure solution, the positional co-ordinates were obtained which were subjected to refinement with their isotropic temperature factors, through SHELXL-97 refinement programme. The R factor dropped to 0.0516 after several cycles of refinement. To reduce R factor to 0.0437, further refinement of the structure was

TABLE 4: Bond length [A] with estimated standard deviation in parenthesis for Captafol

S (1) - N (8)	1.6854 (17)
S (1) - C (10)	1.820 (2)
C1 (1) -C (10)	1.773 (2)
C1 (2) - C (10)	1.767 (2)
C1 (3) - C (11)	1.764 (2)
C1 (4) - C (11)	1.768 (2)
C (1) - C (2)	1.309 (4)
C (1) - C (6)	1.491 (4)
C (2) - H (1)	0.9300
C (3) - C (4)	1.534 (3)
C (3) - H (3A)	0.9700
C (3) - H (3B)	0.9700
C (4) - C (9)	1.502 (3)
C (4) - C (5)	1.533 (3)
C (4) - H (4)	0.9800
C (5) -C (7)	1.517 (3)
C (5) -C (6)	1.543 (3)
C (5) - H (5)	0.9800
C (6) - H (6A)	0.9700
C (7) - H (6B)	0.9700
C (7) - O (13)	1.196 (3)
C (7) - N (8)	1.414 (3)
N (8) -C (9)	1.400 (3)
C (9) -C (12)	1.203 (3)
C (10) - C (11)	1.537 (3)
C (11) -H (11)	0.9800

TABLE 5 : Bond angle [Degree] with estimated standard deviation in parenthesis for Captafol

N(8)-S(1)-C(10)	102.23(9)	C(1)-C(6)-H(6A)	109.3
C(2)-C(1)-C(6)	120.2(2)	C(5)-C(6)-H(6A)	109.3
C(2)-C(1)-H(1)	119.9	C(1)-C(6)-H(6B)	109.3
C(6)-C(1)-H(1)	119.9	C(5)-C(6)-H(6B)	109.3
C(6)-C(1)-C(3)	120.1(2)	H(6A)-C(6)-H(6B)	108.0
C(1)-C(2)-H(2)	120.0	O(13)-C(7)-N(8)	124.32(19)
C(1)-C(2)-H(2)	120.0	O(13)-C(7)-C(5)	127.97(19)
C(2)-C(3)-C(4)	110.7(2)	N(8)-C(7)-C(5)	107.71(17)
C(2)- C(3)- H(3A)	109.5	C(9)-N(8)-C(7)	112.26(17)
C(4)-C(3)-H(3A)	109.5	C(9)-N(8)-S(1)	123.74(14)
C(2)-C(3)-H(3B)	109.5	C(7)-N(8)-S(1)	122.99(14)
C(4)-C(3)-H(3B)	109.5	O(12)-C(9)-N(8)	123.40(19)
H(3A)-C(3)-H(3B)	108.1	O(12)-C(9)-C(4)	127.6(2)
C(9)-C(4)-C(5)	105.33(16)	N(8)-C(9)-C(4)	108.95(17)
C(9)-C(4)-C(3)	109.33(18)	C(11)-C(10)-C1(2)	109.07(15)
C(5)-C(4)-C(3)	114.73(18)	C(11)-C(10)- C1(1)	110.81(15)
C(9)-C(4)-H(4)	109.0	C1(2)-C(10)-C1(1)	109.99(12)
C(5)-C(4)-H(4)	109.0	C(11)-C(10)-S(1)	113.91(15)
C(3)-C(4)-H(4)	109.0	C1(2)-C(10)-S(1)	110.64(11)
C(7)-C(5)-C(4)	105.50(16)	C(1)-C(10)-S(1)	102.26(11)
C(7)-C(5)-C(6)	110.37(19)	C(10)-C(11)-C1(3)	111.09(16)
C(4)-C(5)-C(6)	114.00(19)	C(10)-C(11)-C1(4)	111.84(16)
C(7)-C(5)-H(5)	108.9	C1(3)-C(11)-C1(4)	109.11(13)
C(4)-C(5)-H(5)	108.9	C(10)-C(11)-H(11)	108.2(10)
C(6)-C(5)-H(5)	108.9	C1(3)-C(11)-H(11)	108.2(11)
C(1)-C(6)-C(5)	111.56(19)	C1(4)-C(11)-H(11)	108.2(10)

carried out with individuals' anisotropic temperature factors exponent of the form.

$$-2Pi \wedge 2[h^2a^{*}{}^2U_{11}+..........+2hKa^{*}b^{*}U_{12}]$$

The hydrogen atoms were fixed by geometrical consideration at this stage, but not included in refinement. Refinement of the structure was terminated after two more cycles when all the shifts in Parameter's become much smaller than the corresponding estimated standard deviations. The final R value was 0.0437 for all the 8018 reflections. The final positional and thermal parameters of non-hydrogen atoms are listed in TABLES 2 and 3 respectively.

RESULT AND DISCUSSION

The numbering scheme is shown in figure 1 and ORTEP Diagram is shown in figure 2. The bond lengths and angles are listed in TABLES 4 and 5 respectively. The torsion angles are given in TABLE 6.

It is of interest to see the geometry of Phthalimide group. The C (1)-C (2) bond length is much shorter 1.309(4)Å compared to standard values, whereas the largest bond distance is C (5)-C (6) of 1.543(3)Å But as far as bond angles are concerned, they vary from 110.7(2)° to 120.2(3)°, thus suggest that the ring is compressed as expected. The five-member ring shows usual behavior. The geometry around S (1), C (10) and C(12) appears to be normal as all the lengths are close to single normal bond values and angles are according to the configuration. The N(8)-S(1)-C(1O) angle of
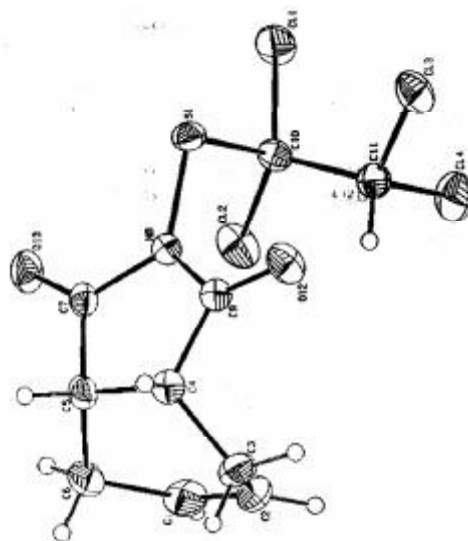

Figure 2 : ORTEP for cis-N-(1,1,2,2-tetrachloroethylthio)-4-cyclohexen1,2-dicarboximide

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TABLE 6 : Torsion angles [deg.] for Captafol

C (6) - C (1) -C (2) -C (3)	0.4 (4)
C (1) - C (2) -C (3) - C (4)	-43.5 (3)
C (2) - C (3) - C (4) - C (9)	42.9 (2)
C (2) - C (3) -C (4) -C (5)	-3.6 (2)
C (9) -C (4) - C (5) -C (7)	-124.22 (19)
C (3) -C (4) -C (5) - C (7)	117.6 (2)
C (9) -C (4) -C (5) - C (6)	-3.0(3)
C (3) -C (4)-C (5) - C (6)	41.7(4)
C (2) -C (1)-C (6) - C (5)	80.8 (3)
C (7) -C (5)- C (6)- C (1)	-37.7 (3)
C (4)- C (5)- C (6)- C (1)	-175.1 (2)
C (4) -C (5)- C (7)- C (13)	61.3 (3)
C (6) -C (5) -C (7)- C (13)	5.0 (2)
C (4) -C (5) -C (7)- N (8)	-118.56 (19)
C (6) -C (5) -C (7) -N (8)	175.5 (2)
O (13)-C (7) - N (8) -C (9)	-4.7 (2)
C (5) - C (7) - N (8) -C (9)	6.6 (3)
O (13) -C (7) -N (8) -S (1)	-173.57 (13)
C (5) - C (7) - N (8) - S (1)	89.59 (17)
C (10) -S (1) - N (8) - C (9)	-102.77 (16)
C (10) -S (1) - N (8) -C (7)	-178.7 (2)
C (7) - N (8) - C (9) - O (12)	-9.9 (3)
S (1) - N (8) - C (9) - O (12)	2.3 (2)
C (7) - N (8) - C (9) - C (4)	171.09 (13)
S (1) - N (8) - C (9) - C (4)	-177.09 (13)
C (5) -C (4) - C (9) - O (12)	-54 -.0 (3)
C (5) -C (4) -C (9) - N (8)	1.0 (2)
C (3) -C (4) -C (9) - N (8)	124.94 (19)
N (8) -S (1) -C (10)-C (11)	-74.94 (16)
N (8) -S (1) -C (10) -C1 (2)	48.35 (13)
N (8) -S (1) -C (10) -C1 (1)	165.46 (10)
C1 (2)-C (10) -C (11) -C1 (3)	178.88 (11)
C1 (1) -C (10) -C (11) -C1(3)	57.67 (19)
S (1) -C (10) - C (11) - C1 (3)	-56.97 (19)
C1 (2) -C (10)-C (11) -C1(4)	56.70 (18)
C1 (1) -C (10) -C (11) - C1(4)	-64.52 (19)
S (1) -C (10) -C (11) -C1 (4)	-179.16 (11)

102.2(9)° shows that the chain is almost right angle to phthalimide group. The angle of twist between phthalimide group and remaining chain N (8)-S (1)-C (10)-C (11) is of -74.9(2)°. The phthalimide group appears to be planner, as we calculated mean planes using Blow's method. If we look to the angles between different planes, it appears that the molecule is highly twisted and folded.

Molecular packing

The molecules are stacked, along c-axis and held firmly through non-bonded contacts, because there does not exist any hydrogen bond. The groups are stacked in such a way that they generate significant Vander-Walls interaction between them.

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X-ray crystallographic studies on tetra methyl thiram disulphide or bis (dimethyl thiocarbamoyl) disulphide

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ABSTRACT

It has been observed that some of the fungicides are loosing their effects and becoming resistant to them. Analogous compounds can be designed as substitute, if their structures are known. A rational approach to test these fungicides is to know the three dimensional structure of these compounds and macromolecular receptor sites as well as their molecular complex The structures of these compounds can be obtained by X-ray diffraction method in crystalline form and they will invariably be similar to their structure in solutions.The composition of Tetra methyl thiram disulphide or bis (dim ethyl thiocarbamoyl) disulphide crystals are confirmed by comparing the infra-red spectra of the two components. The Unit cell parameters are a=6.91 30(10)Å,b=6.9250(10)Å c=11.8020(10)Å the space group is P1.   © 2008 Trade Science Inc. - INDIA

INTRODUCTION

Thiram is the coined name for tetramethyl thiram disulphide or bis (dimethyl thiocarbamoyl) disuiphide. The molecular weight of thiram is 240.4. It is white coloured substance which is essentially insoluble in water slightly soluble in alcohol and ether and completely soluble in acetone and chloroform. It is unstable in the presence of acids. Being carbomate fungicidal compound thiram is toxic if consumed orally.As a part of research, X-ray crystallographic analysis of this fungicidal compound was carried out to study the effect of substituents on the molecular geometry and conformation of fungicidal compound with the main object to understand their biological activity.

EXPERIMENTAL

It was developed by E.l. du Pont de Nemours and Co. in 1931. The crystals were grown at 20^0 from its solution in acetone by slow evaporation method. The unit cell parameters were determined directly by automatic computerized 4 Enraf Nonious CAD-4 diffractometer in ω-2θ scan mode using Nickel filtered CuKa, radiation with range of 3.8 to 69.94°. These data showed a=6.9130(10)Å, b=6.9250(10.)Å, c=11.8020(10)Å. The space group is P1. The density of the crystal was measured by floatation method, in the mixture of benzene and carbon tetrachloride at room temp. Its calculated density is 1.469 mg/m^3 and measured density is 1.454 mg/m^3

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Data collection and structure solution

The three dimensional intensity data were collected on a computerized automatic 4-circled CAD-4 Enraf-Nonious diffractometer using Nickel filtered CuKa(1.5418A) radiation at Deptt. of Biophysics AIIMS New Delhi. All these data were corrected for Lorentz and polarization effect, but no absorption correction was applied. The total number of reflection were 2247. The unique reflections corresponding to intensity limit were 2064. The hkl values varies from 0<=h<=8, -8<=k<=8, -14<=k=14 respectively. The crystal structure was solved using the SHELXS-97. The ORTEP Diagram is shown in figure 1 and packing diagram is shown in figure 2.

Refinement

For determination of structure of the crystal VAX machine using SHELXS-97° was used. In the beginning all the non-hydrogen atom'. are located The coordinates thus obtained are fed to SHELXL-97 for refinement The first three cycles of isotropic refinement gave on R value of 16.21%. To reduce R to 14.25% given form of refinement of structure with individual an isotropic temp. factors is in exponential form was implemented

2P^[1+ +2hKa*b*U12]

All the hydrogen atoms were fixed geometrically and not included in the refinement. The refinement was terminated when all the shifts indicated for parameters of non-hydrogen atoms were less than the corresponding standard deviation. The final R index was 13.46% for all the observed reflections.

RESULT AND DISCUSSION

The molecule consists of two diethyl, dithocarbamato moieties connected through S(2)-S(3). The S(2)-S(3) bond length is of 2.0077(16)A. The conformation of the molecule is similar to that of thiram monosulphide and even more so that of several sulphides This is also a good ideal system for the study of deformation density. For the reasons, the molecule contains various C-N, C-S, S-S bonds. If the molecule is divide along S(2)-S(3) bond in two equal halves, it shows symmetry in parameters. Each of the two dimethyldithiocarbamato crystals approximately plan-
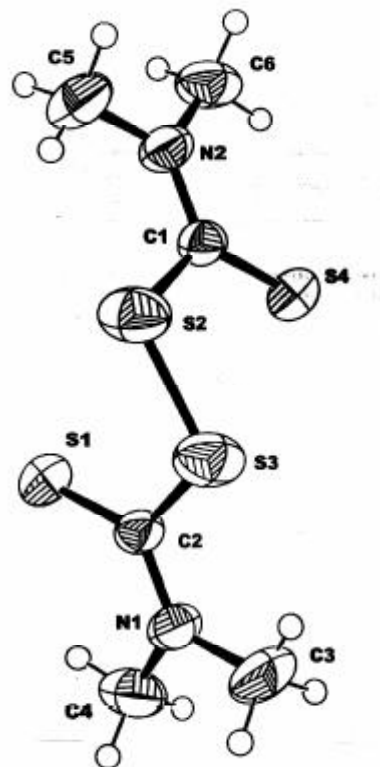

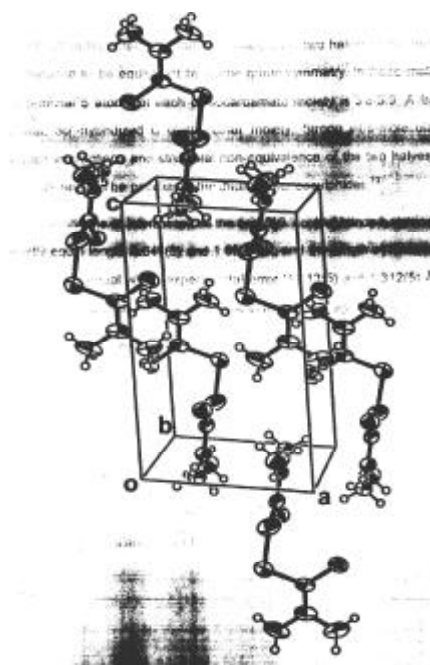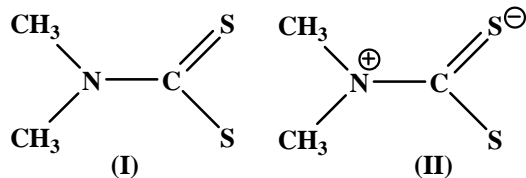**Figure 1: ORTEP drawing at 50% probability level**

Figure 2 : The moleculer pacing seen down b-axis

ner. The dihedral angle around S(2)-S(3) bond is -88.38(17)°. The length of the C-N and terminal C-S bonds are intermediate between the values expected

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(I)   (II)

for single and double bonds, indicating that the canonical form II makes a substantial contribution of the structure.

The high double bond character of these bonds also affects the C-N stretching frequencies of the infrared spectrum, which were shifted considerable towards higher frequency from the normal C-N region.

It may be. added that in tetramethyithiram disulphide the two halves of the molecules are required to be equivalent by space group-symmetry. In these molecules, the terminal 5 atoms of each dithiocarbamato crystal is 3.8-3.9Å from the central, Sp C of the other moiety. Strong intra-molecular non- bonded interactions and structural non-equivalence of the two halves of the molecule seen to be peculiar of the thiuram monoscul phides. It is of interest to see the two C-S 'double' bonds happen to have exactly equal length 1.648(3)Å and 1.652(4)Å, and the length of two central C- N bonds are equal [within experimental error] and 1.312(5)Å. Also the lengths of the four N-CH bonds are in reasonable agreement. It is well known that among the several factors affecting the values of bond angles is Sp Sp hybridized centres. An important role is played by the repulsions between bonding (non-bonding) electron pairs in the valence shell of the central atom. In a survey of the molecular geometries of urea derivatives the value of the N-C-N angles has been found tc increase regularly with the length of opposite bond. C-O. This results is easily accounted for in terms of varying repulsions between bonding electron pairs in valance cell of C.

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