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# Structural Investigations of Ylides. XII. Crystal and Molecular Structure of *N*-(*p*-Nitrobenzoyl)-2-iminophenyldimethylsulphur(IV), C<sub>15</sub>H<sub>14</sub>N<sub>2</sub>O<sub>3</sub>S

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The structure of the title compound has been determined from 2209 independent diffractometer data, and refined by least squares to a final R of 0.040. The crystals are orthorhombic, space group Pbca, with Z=8, a=25.971, b=8.822, c=13.310 Å. The S<sup>+</sup>-C(ar) and C(ar)-N<sup>-</sup> lengths are 1.772 (2) and 1.385 (3) Å respectively, which in conjunction with the observed conformation suggest little delocalizing interaction between the sulphonium and imine species via the intervening aromatic ring.

#### Introduction

We have recently discussed the structure of the sulphonium-imine ylide (I), comparing the main features of the ylidic geometry with other available examples (Cameron, Duncanson & Morris, 1976). We now describe the structure of the unusual compound (II), which may be regarded as a structural variant of ylides of the type represented by (I), in which an aromatic ring is interposed between the sulphonium and imine species.



Whereas in the simpler sulphonium-imines [e.g. (I)], delocalization in the  $S^+-N^-$  moiety is thought to take place by interaction between the vacant S *d*orbitals and the lone pair(s) of electrons on the N atom, in (II) such a simple interaction is impossible. However, it is possible to write several canonical forms [(IIIa)  $\leftrightarrow$  (IIIb)  $\leftrightarrow$  (IIIc)] showing interaction between the aromatic system and the imine atom, with the additional possibility of interaction between the vacant S *d*-orbitals and the perturbed aromatic system.

#### Experimental

## Crystal data

*N*-(*p*-Nitrobenzoyl)-2-iminophenyldimethylsulphur(IV), C<sub>15</sub>H<sub>14</sub>N<sub>2</sub>O<sub>3</sub>S, *M*=302·4. Orthorhombic, a=25.971, b=8.822, c=13.310 Å, U=3046.7 Å<sup>3</sup>,  $D_m=1.31$  (by flotation), Z=8,  $D_c=1.32$  g cm<sup>-3</sup>, F(000)=1264. Space group *Pbca* ( $D_{2h}^{15}$ , No. 61). Mo K $\alpha$  radiation,  $\lambda = 0.7107$  Å,  $\mu$ (Mo K $\alpha$ ) = 2.23 cm<sup>-1</sup>.

## Crystallographic measurements

The cell parameters were initially determined photographically and were refined by least squares prior to the diffractometer data collection. Intensity measurements were made on a Hilger & Watts Y290 diffractometer with a small crystal exposed to graphitemonochromated Mo radiation and with the  $\theta, \omega$ -scan technique to measure the intensities of 2209 independent reflexions  $[I > 3\sigma_I, \sigma_I = \gamma(I + B_1 + B_2)]$  in the range  $2\theta = 0 \rightarrow 60^\circ$ . The intensities were corrected for Lorentz and polarization factors, but not for absorption.

#### Structure determination and refinement

The structure was determined by direct methods with the multi-symbolic technique, triplet relationships being derived for those 256 reflexions with  $|E| \ge 1.40$ , and a solution for the 60 strongest reflexions being obtained with relationships of probability  $\ge 0.8$ . 231 phases were then determined from this solution, and the resulting *E* map yielded all but four of the non-hydrogen atom positions. A subsequent structure factor and electron density calculation revealed the remaining four atoms.

Refinement by least squares was terminated when R was 0.040. Details of the refinement are presented in Table 1, from which it will be seen that some effort was devoted to examining subsets of the data. This was undertaken to investigate marked anisotropy with high values of thermal parameters for the nitro group and neighbouring ring atoms. It was eventually concluded that the anomalous thermal motion of the nitrobenzoyl group is a property of the whole data set, and this will be discussed later in relation to the molecular packing.

All H atoms were located from difference syntheses, and parameters for these atoms were refined in later cycles. The weighting scheme was  $w = (A + B|F_o| + C|F_o|^2)^{-1}$ , the final values of the parameters A, B and C being 2.701, -0.241 and 0.0105 respectively. On

#### Table 1. Course of refinement

Cycles	Parameters refined	Final R	Final R'
1-5	$x, y, z, U_{iso}$ for S, O, N and C; scale factor. Unit weights, full matrix.	0.130	0.0196
6-11	$x, y, z, U_{ij}$ for S, O, N and C; scale factor. Contributions for four H atoms included but not refined. Unit weights, full matrix.	0.070	0.0077
12	As previously, but with corrections to strong reflexions for counter-saturation errors.	0.063	0.0055
13	As previously, but with all reflexions with $ \Delta  > 10.0$ excluded (10 reflexions).		
	No major shifts in parameters.		
14	As previously, but with all reflexions with $\sin \theta/\lambda \le 0.25$ ( $2\theta \simeq 20^\circ$ ) excluded (187 reflexions).	0.024	0.0035
	No major shifts in parameters.		
15	As previously, but with all reflexions with $\sin \theta / \lambda \le 0.45$ ( $2\theta \simeq 37^{\circ}$ ) excluded (970 reflexions).	0.043	0.0025
	Some shifts in thermal parameters, but no major changes.		
16–18	$x, y, z, U_{lj}$ for S, O, N and C; $x, y, z, U_{lso}$ for H (14 H atoms); scale factor.	0.040	0.0014
	Unit weights, blocked matrix.		
19–22	$x, y, z, U_{ij}$ for S, O, N and C; $x, y, z, U_{iso}$ for H; scale factor.	0.040	0.0035
	Weighting scheme applied, blocked matrix.		

convergence, evaluation of a difference synthesis revealed no errors in the structure, and the refinement was considered complete.\*

Final coordinates and thermal parameters are listed in Table 2, details of the molecular geometry in Table 3. The estimated standard deviations quoted in Tables 2 and 3 derive from the least-squares calculations and should be regarded as minimum values. Fig. 1 shows a view of one molecule and defines the atomic numbering, while the molecular packing viewed down **b** is illustrated in Fig. 2.

#### Discussion

Although the conformation adopted by (II) is more or less extended, there are certain deviations from planarity which are significant in relation to the possibility of extended delocalization in the molecule. Foremost among these is the torsion about N(1)-C(2), such that the plane of the sulphonium-substituted aromatic ring is inclined at an angle of  $37.8^{\circ}$  to the plane of the carbonyl-imine system. This feature suggests minimal delocalization of the negative charge into the aromatic system [as in (III)], a conclusion which is supported by the length of N(1)-C(2) [1.385 (3) Å] which is

\* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31671 (16 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.



Fig. 1. A view of one molecule showing the atomic numbering.

significantly longer than the N–C bond in free pyridine  $[1\cdot340(5) \text{ Å}]$  (Bak, Hansen & Rastrup-Andersen, 1954). In further support, S<sup>+</sup>–C(1)  $[1\cdot772(2) \text{ Å}]$  is comparable with S<sup>+</sup>–C(ar) in (IV)  $[1\cdot769(8) \text{ and } 1\cdot799(8) \text{ Å}]$  (Kálmán, Duffin & Kucsman, 1971), and longer than S<sup>+</sup>–C in (V)  $[1\cdot730(8) \text{ Å}]$  (Christensen & Witmore,

# Table 2. Fractional coordinates and thermal parameters

(a) Fractional coordinates

	x	У	Z
S	-0.03927(2)	0.35228(6)	0.45909(4)
N(1)	0.01670 (7)	0.12640(23)	0.37049 (14)
N(2)	0.25542 (15)	0.10654 (87)	0.48720 (44)
O(1)	0.05267 (7)	0.05156 (27)	0.21956 (13)
O(2)	0.29145(12)	0·07771 (122)	0.43575 (41)
O(3)	0.25981(13)	0.13269 (78)	0.57423 (37)
C(1)	- 0·06899 (8)	0.21633 (27)	0·38015 (15)
C(2)	-0.03394(8)	0.11201 (27)	0.33919 (15)
C(3)	-0.05485(11)	-0.00220 (33)	0.27799 (18)
C(4)	-0.10712(11)	<i>−</i> 0·00636 (36)	0.25864 (20)
C(5)	-0.14004(11)	0.10072 (39)	0.29672 (23)
C(6)	-0.12149(10)	0.21321 (35)	0.35910 (20)
C(7)	0.10753 (9)	0.09538 (30)	0.35806 (18)
C(8)	0.11382 (11)	0.12990 (33)	0.45911 (21)
C(9)	0.16264 (13)	0.13595 (46)	0.50121 (28)
C(10)	0.20383 (12)	0.10327 (59)	0.44173 (35)
C(11)	0.19908 (13)	0.06907 (84)	0.34125 (38)
C(12)	0.15054 (12)	0.06536 (60)	0.30053 (32)
C(13)	0.05496 (9)	0.08832 (29)	0.30976 (17)
C(14)	-0.03174(13)	0.25035 (37)	0.57472 (21)
C(15)	-0.08967(11)	0.48056(37)	0.49308 (23)

(b) H atom fractional coordinates and isotropic thermal parameters  $(Å^2 \times 10^3)$ 

-				
	x	У	Z	$U_{iso}$
H(3)	-0.035(1)	-0·078 (4)	0.256 (2)	60 (8)
H(4)	-0.121(1)	-0.086(4)	0.219(2)	72 (9)
H(5)	-0.177(1)	0.100 (3)	0.286 (2)	63 (8)
H(6)	-0.143(1)	0.284 (4)	0.393 (2)	66 (9)
H(8)	0.087 (1)	0.150 (3)	0.496 (2)	53 (8)
H(9)	0.164 (1)	0.157 (4)	0.568 (3)	79 (11)
H(11)	0.227 (2)	0.045 (6)	0.302(4)	138 (17)
H(12)	0.145 (1)	0.037 (5)	0.226(3)	112 (14)
H(141)	-0·012 (1)	0.159 (4)	0.559 (2)	77 (10)
H(142)	-0·063 (1)	0.226 (4)	0.597 (2)	71 (9)
H(143)	-0.013(1)	0.314 (4)	0.620(3)	82 (10)
H(151)	- <b>0</b> ·117 (1)	0.424 (3)	0.520 (2)	58 (8)
H(152)	-0·075 (1)	0.548 (3)	0.543 (2)	60 (8)
H(153)	-0.098(1)	0.530 (4)	0.431(3)	69 (9)

Table 2 (cont.)

- · · · · · · · · · · · · · · ·	r		<b>v</b>	,	
$U_{11}$	$U_{22}$	$U_{33}$	$U_{12}$	$U_{13}$	$U_{23}$
382	442	369	0	16	16
413	493	377	58	-9	- 48
488	3298	1556	-167	-268	- 48
572	982	434	98	22	- 201
445	6783	2037	282	-214	- 530
828	3867	1496	-413	- 545	33
398	455	365	-41	-7	- 8
453	472	298	-655	-10	25
602	511	388	66	8	-20
653	671	458	-212	- 68	- 49
441	849	612	-175	- 87	-25
404	670	565	- 2	-27	-28
425	510	524	38	-11	-4
469	692	554	- 51	- 49	61
605	1003	697	-173	-192	92
401	1478	1154	- 39	- 174	78
425	2417	1068	140	29	- 395
497	1617	796	154	38	- 291
463	452	414	50	- 8	- 16
679	573	372	8	-40	25
510	611	489	100	82	- 36
timated sta	andard d	leviations	$(Å^2 \times 10)$	<sup>(4</sup> )	
$U_{11}$	$U_{22}$	$U_{33}$	$U_{12}$	$U_{13}$	$U_{23}$
4	4	4	2	2	2
14	45	25	21	16	29
17	70	30	32	16	40
14	14	14	14	14	16
	$U_{11}$ 382 413 488 572 445 828 398 453 602 653 441 404 425 469 605 401 425 497 463 679 510 timated sta $U_{11}$ 4 14 17 14	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$

1969) and (VI) [1.707 (4) Å] (Christensen & Thom, 1971). There would thus appear to be negligible delocalizing interaction between the sulphonium and aromatic species. Possibly conflicting with these con-

### Table 3. Interatomic distances and angles

(a) Bonded distances (Å)

S———C(1) $1.772$ (2)	C(5) - C(6) = 1.381(4)
S - C(14) = 1.793(3)	C(6) - C(1) = 1.392(3)
S - C(15) - 1.789(3)	C(7) - C(8) = 1.389(4)
N(1) - C(2) = 1.385(3)	C(8) - C(9) = 1.387(4)
N(1) - C(13) = 1.324(3)	C(9) - C(10) = 1.362(5)
O(1) - C(13) + 1.245(3)	C(10) - C(11) = 1.377(7)
C(13) - C(7) = 1.511(3)	C(11) - C(12) + 1.373(5)
C(1) - C(2) = 1.405(3)	C(12) - C(7) 1.380 (4)
C(2) - C(3) = 1.405(3)	C(10) - N(2) = 1.470(5)
C(3) - C(4) = 1.382(4)	N(2) - O(2) + 1.187(7)
C(4) - C(5) = 1.371(4)	N(2) - O(3) = 1.187(8)
Mean	Mean
C-H (methyl) 0.95 (3)	C-H (arvl) 0.94 (4)
(b) Interbond angles (°)	
C(1)— $S$ — $C(14)$ 102·5 (1)	C(4) - C(5) - C(6) = 120.0 (2)
C(1) = S = C(15) 105.0(1)	C(5) - C(6) - C(1) = 118.5(2)
C(14)-SC(15) 100.4 (1)	C(13)-C(7)-C(8) 121.8 (2)
SC(1)-C(2) $113.0(1)$	C(13)-C(7)-C(12) 119.2 (2)
S - C(1) - C(6) = 124.0(1)	C(7)-C(8)-C(9) 120.5 (2)
C(1)-C(2)-N(1) 116.0 (1)	C(8) - C(9) - C(10)  118.4  (3)
C(3) - C(2) - N(1)  127.4  (2)	C(9) - C(10) - C(11)  122.7  (3)
C(2) - N(1) - C(13)  120.4  (1)	C(10)-C(11)-C(12) 118.1 (3)
$N(1) - C(13) - O(1)  128 \cdot 2 (1)$	C(11)-C(12)-C(7) 121.3 (3)
N(1)-C(13)-C(7) 114·1 (2)	$C(9) - C(10) - N(2) = 118 \cdot 2 (3)$
O(1) - C(13) - C(7) = 117.7(2)	C(11)-C(10)-N(2) 119.1 (3)
C(6) - C(1) - C(2) = 122.9(2)	C(10)-N(2)-O(2) 118.5 (4)
	(1,1,1,1,1,1,1,1,1,1,1,1,1,1,1,1,1,1,1,

 $\begin{array}{c} C(10) - N(2) - O(3) & 119 \cdot 6 \ (3) \\ O(2) - N(2) - O(3) & 121 \cdot 9 \ (4) \\ C(12) - C(7) - C(8) & 119 \cdot 0 \ (2) \end{array}$ 

116.4 (2)

120.5 (2)

121.7 (2)

C(1) - C(2) - C(3)

-C(3)-

C(2)

-C(4)

-C(4)-C(5)

|--|

(c) Intramolecular non-bonded distances (Å)				
SN(1)	2.73	$O(1)\cdots C(3) = 2$	93	
$N(1) \cdots C(14)$	3.19	$O(1) \cdots C(12) = 2$	76	
$N(1) \cdots O(1)$	2.31	$O(2) \cdots C(11) 2$	71	
$N(1) \cdots C(8)$	2·78	$O(3) \cdots C(9) = 2$	70	
$O(1) \cdots C(2)$	<b>2</b> ·81	$O(2) \cdots O(3) = 2$	08	
(d) Intermolecular di	stances (Å)			
$N(1) \cdots C(14)^i$	3.42	$C(14) \cdot \cdot \cdot C(2)^{iii}$	3.72	
$C(9) \cdot \cdot \cdot C(4)^i$	3.69	$C(14) \cdots C(3)^{iii}$	3.55	
$C(9) \cdot \cdot \cdot C(5)^i$	3.45	$C(15) \cdots C(4)^{iii}$	3.57	
$C(14) \cdots O(1)^i$	3.86	$S \cdots O(1)^{iv}$	<b>2</b> ∙98	
$C(14) \cdots C(13)^i$	3.41	$C(1) \dots O(1)^{iv}$	3.27	
$C(14) \cdots C(3)^i$	3.70	$C(6) \cdots O(1)^{iv}$	3.63	
$C(14) \cdots C(7)^i$	3.74	$C(15) \cdots O(1)^{iv}$	3.05	
S·····S <sup>ii</sup>	3.48	$O(2) \cdots C(5)^{v}$	3.58	
$\mathbf{S} \cdots \mathbf{C} (15)^{i1}$	3.71	$O(2) \cdots C(15)^{vi}$	3.27	
$C(15) \cdots C(8)^{ii}$	3.55	$O(3) \cdots C(6)^{vi}$	3.48	
$C(14) \cdots O(1)^{iii}$	3.40			

Roman numeral superscripts refer to the following equivalent positions relative to a molecule at (x, y, z):

(i)	-x	-y	1 - z	(ii)	-x	1-y	1 - z
(iii)	x	$\frac{1}{2} - y$	$\frac{1}{2} + z$	(iv)	-x	$\frac{1}{2} + y$	$\frac{1}{2} - z$
(v)	$\frac{1}{2} + x$	У	$\frac{1}{2} - Z$	(vi)	$\frac{1}{2} + x$	$\frac{1}{2} - y$	1 - z

(e) Torsion angles (°)

C(2) - N(1) - C(13) - O(1)	7.5
C(2) - N(1) - C(13) - C(7)	-174·2
N(1) - C(13) - C(7) - C(8)	3.8
N(1) - C(13) - C(7) - C(12)	- 176.8
O(1) - C(13) - C(7) - C(8)	-177.6
O(1) - C(13) - C(7) - C(12)	1.8
N(1) - C(2) - C(1) - S	3.3
N(1) - C(2) - C(1) - C(6)	-177.6
N(1) - C(2) - C(3) - C(4)	175.7
C(2) - C(1) - S - C(14)	- 80.3
C(2) - C(1) - S - C(15)	175.2
C(6) - C(1) - S - C(14)	100.6
C(6) - C(1) - S - C(15)	-4.0
C(9) - C(10) - N(2) - O(2)	179.8
C(9) - C(10) - N(2) - O(3)	2.3
C(11)-C(10)-N(2)-O(2)	- 0.9
C(11)-C(10)-N(2)-O(3)	178-4
C(13)-N(1)-C(2)-C(1)	- 149.5
C(13)-N(1)-C(2)-C(3)	36.3
Mean e.s.d. 0.3°	

(f) Least-squares planes

X, Y and Z are orthogonal coordinates in Å. An asterisk denotes atoms used to define plane.

(1) Equations of planes

(I)	-0.1430X - 0.5847Y + 0.7986Z + 3.1678 = 0
(II)	0.0736X + 0.9699Y - 0.2322Z - 0.0846 = 0
(III)	-0.0917X - 0.9578Y + 0.2723Z + 0.2374 = 0

(2) Deviations of atoms from the planes (Å)

(I)	S	0.041	N(1)	0.056	C(13)	-0.535
	C(15)	-0.072	$\mathbf{C}(1)$	0.013*	C(2)	-0.014*
	C(3)	0.002*	C(4)	0.012*	C(5)	-0.013*
	C(6)	0.001*				
(II)	N(1)	0.053	O(1)	-0.052	C(13)	-0.012
	N(2)	-0.022	O(2)	-0.040	O(3)	-0.058
	C(7)	-0.001*	C(8)	-0.006*	C(9)	0.010*
	C(10)	-0.008*	C(11)	-0.001*	C(12)	0.003*
(III)	C(7)	-0.002*				
	O(1)	-0.003*				

(3) Dihedral angles between planes: (I) and (II) 139.7°; (II) and (III)  $2 \cdot 6^\circ$ ; (I) and (III)  $37 \cdot 8^\circ$ .



Fig. 2. The molecular packing viewed down b.

clusions is the perceptible lengthening of C(1)-C(2) [1.405 (3) Å] and C(2)-C(3) [1.405 (3) Å] in relation to the other values within this ring. However, if significant, the effect is slight and could well be a reflexion of the *ortho* substitution rather than evidence for extended delocalization.





C(2)-

C(6) -4·0°



N(1)-C(2) projection







Fig. 4. The overlap of adjacent nitrobenzene groups viewed (a) perpendicular to **b**, and (b) parallel to **b**.

It would appear, therefore, that the major delocalization of the negative charge should take place into the carbonyl group, and in this respect we note the short N(1)-C(13) [1·324 (3) Å], indicative of high double-bond character, in conjunction with the length

of the carbonyl bond [1.245 (3) Å]. Moreover, the  $C(2)-N(1)^{-}-C(13)=O(1)$  grouping is close to planarity [torsion angle 7.5°]. Since we have suggested minimal delocalization of the negative charge into the sulphonium-aromatic system, there should therefore be a similarity between (II) and (VII) (Cameron, Hair & Morris, 1972), in which there is no delocalization between the 'onium and imine species, in contrast to (I) where some delocalization across the  $S^+-N^$ bond is postulated. Thus in (VII) the  $N^--C$  and C=O lengths are 1.313 (6) and 1.243 (5) Å respectively, while the corresponding values in (1) are 1.344 (3) and 1.246 (3) Å. In particular, therefore, there is an apparent progression of values for the  $N^--C(O)$  bonds in the three molecules, which may well reflect the degree of delocalization between the 'onium and imine groups in each case.

The major conformational features of (II) are illustrated by the Newman projections in Fig. 3. The 36° torsion about N(1)–C(2) may well arise from steric interactions involving the carbonyl group, the sulphonium-substituted aromatic ring and the sulphonium group itself  $[O(1)\cdots C(3) 2.93 \text{ Å}, S^+\cdots N(1) 2.73 \text{ Å}]$ . As a further possible consequence of such interactions, the valence angle at N(1) [120.4 (1)°] is considerably larger than the corresponding angles in (I) [110.0 (1)°] and similar molecules, while the value of 128.2 (1)° for N(1)–C(13)–O(1) is similar to that observed elsewhere.

One interesting feature of the crystal structure of (II) is related to the large thermal motion of the terminal nitro-aromatic group, and is illustrated by the thermal ellipsoid plots in Fig. 4, which shows the over-

lap of pairs of **b**-glide related molecules viewed parallel to and perpendicular to b. The nature and direction of the motion thus viewed, suggests a tendency for overlapped nitrobenzene groups to associate closely in pairs by the out-of-phase flexing of adjacent molecules, to give rise to stacks of pairs of associated molecules extending throughout the crystal. Consideration of the r.m.s. amplitude of vibration in relation to the b spacing, indicates a closest possible approach of overlapped nitro groups of the order of  $3 \cdot 2 - 3 \cdot 3$  Å, such that the possibility of charge-transfer interaction cannot be excluded. However, it must be noted that although the colour of the crystals is intense orange, this colour not only persists in solution, but is solvent invariant. Whatever the exact nature of the above interaction, it would seem to be the dominant feature of the crystal packing, although several short intermolecular contacts involving the sulphonium group  $[S \cdots S 3.48, S \cdots O(1) 2.98 \text{ Å}]$  must be noted.

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# The Crystal Structure of the Methanesulphonamide of Piperidine-4-spiro-1'-(2',5'-dioxolane) (C<sub>8</sub>H<sub>15</sub>NSO<sub>4</sub>)

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Crystals of the methanesulphonamide of piperidine-4-spiro-1'-(2',5'-dioxolane) are monoclinic, space group  $P2_1/c$  with a = 10.373 (2), b = 5.449 (1), c = 18.049 (4) Å and  $\beta = 91.87$  (1)°. The structure has been refined to R = 0.076 for 2936 independent reflexions measured on an automatic four-circle diffractometer. The piperidine ring has a chair conformation and the S-N bond length of 1.634 Å shows a certain amount of double-bond character.

#### Introduction

It is difficult to carry out the conformational analysis of N-methylsulphonyl compounds by liquid phase methods (dipole moments and NMR) owing to (a) the flattening at the nitrogen atom involving a not completely tetrahedral arrangement of substituents and (b) the possible existence of rotamers around the N-S bond. For these reasons Dr Moreno Mañas of Universidad Autonoma de Barcelona, suggested