References

FURUYA, T., IWANAMI, S., TAKENAKA, A. & SASADA, Y. (1982). *Bull. Chem. Soc. Jpn*, **55**, 2321–2326.

FURUYA, T., IWANAMI, S., TAKENAKA, A. & SASADA, Y. (1985). *Acta Cryst.* **C41**, 1507–1509.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 71–151. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

IWANAMI, S., TAKASHIMA, M., HIRATA, Y., HASEGAWA, O. & USUDA, S. (1981). *J. Med. Chem.* **24**, 1224–1230.

LEE, B. & RICHARD, F. M. (1971). *J. Mol. Biol.* **55**, 379–400.

McCANDLISH, L. E., STOUT, G. H. & ANDREWS, L. C. (1975). *Acta Cryst.* **A31**, 245–249.

MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

SHELDRICK, G. M. (1976). *SHELX76.* Program for crystal structure determination. Univ. of Cambridge, England.

Structure of N-(2-Nitrophenylthio)-S,S-diphenylsulfoximide. Intramolecular S···O Interaction

BY FUJIKO IWASAKI

Department of Materials Science, University of Electro-Communications, Chofu-shi, Tokyo 182, Japan

Abstract. $C_{18}H_{14}N_2O_3S_2$, $M_r = 370.44$, monoclinic, $C2/c$, $a = 27.067$ (4), $b = 8.483$ (1), $c = 14.755$ (2) Å, $\beta = 92.20$ (1)°, $V = 3385.3$ (8) Å^3, $Z = 8$, $D_x = 1.454$ Mg m^{-3}, Cu$K\alpha_1$, $\lambda = 1.54051$ Å, $\mu = 2.93$ mm^{-1}, $F(000) = 1536$, $T = 296$ K, $R = 0.039$ for 2669 observed reflexions. The dimensions of the S-sulfoximido group are similar to those of other N-sulfoximides. The bonds about the tetracoordinated sulfur S^{VI} form a distorted tetrahedron with an NSO angle of 122.7 (1)°. The 2-nitrophenylthio group lies almost in one plane and associated with this planarity a short intramolecular S···O contact of 2.532 (2) Å is observed. The angle O···S–N is 175.0 (1)°.

Introduction. As a part of the systematic investigation of sulfoximides the title compound was synthesized by Akutagawa, Furukawa & Oae (1984). This is the first case in which the imido nitrogen is attached to sulfur; all other sulfoximides so far reported by X-ray analysis are those with the imido nitrogen attached to N. In order to investigate the structural character of this novel compound an X-ray analysis was undertaken.

Experimental. Orange plates, $0.30 \times 0.40 \times 0.10$ mm, unit-cell parameters by least squares from 25 reflexions $(65 < 2\theta < 73°)$, Rigaku AFC4 diffractometer, graphite monochromator, $2 < 2\theta < 130°$, $h = 0$–31, $k = 0$–9, $l = -17$–17, ω–2θ scan, scan range $\Delta\omega = 1.0° + 0.5° \tan\theta$, scan speed 4° min^{-1} in 2θ, three reflexions monitored (006, 12,0,0, 333) every 50 reflexions, variations within 0.5%. 2915 measured, 2853 unique, 2669 observed $[|F_o| \geq 3\sigma(F)]$ reflexions.

Structure solved by *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). All non-H atoms obtained from E map. H atoms found from difference map. Block-diagonal least squares with anisotropic temperature factors for non-H and isotropic for H. $\sum w(|F_c| - k^{-1}|F_o|)^2$ minimized. $w = 0.5$ if $|F_o| < 7.0$, $w = 1.0$ if $7.0 \leq |F_o| \leq 28.0$, $w = (28/|F|)^2$ if $|F_o| > 28.0$. Absorption correction applied numerically. Extinction parameter $g = 4 \times 10^{-7}$. $\Delta\rho$(max.) $= 0.20$ e Å^{-3} [between S(1) and C(1)], Δ/σ(max.) $= 0.14$. $R = 0.039$, $wR = 0.050$, $S = 1.098$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). All computations performed on HITAC M180 computer of the Data Processing Center of the University with programs *UNICS*III (Sakurai & Kobayashi, 1979), *MULTAN78* and *ORTEP*II (Johnson, 1971). The final atomic parameters are given in Table 1.*

Discussion. The molecular structure with the atom numbering is shown in Fig. 1. Bond distances and angles are listed in Table 2. All sulfoximides so far reported by X-ray analysis are those with the imido nitrogen attached to N (Andreetti, Bocelli & Sgarabotto, 1975, 1977; Andreetti, Bocelli, Coghi & Sgarabotto, 1974, 1975; Cabezuelo, Foces-Foces,

---

* Lists of structure factors, anisotropic temperature factors and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42484 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional parameters* $(\times 10^5)$ *and equivalent isotropic temperature factors* (Å^2) *for non-H atoms*

$$B_{eq} = \tfrac{4}{3}\Sigma_i \Sigma_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j.$$

	x	y	z	B_{eq}
S(1)	31795 (2)	32298 (7)	54970 (3)	3·6
S(2)	41037 (2)	23199 (6)	60985 (3)	3·1
N(1)	36386 (7)	18428 (21)	55240 (12)	3·9
N(2)	24251 (7)	53726 (22)	44896 (13)	4·2
O(1)	44132 (7)	36311 (19)	58660 (12)	4·9
O(21)	24817 (6)	52068 (23)	53157 (12)	5·6
O(22)	21022 (7)	62085 (24)	41395 (15)	6·4
C(1)	31072 (7)	35028 (22)	43207 (12)	3·0
C(2)	27546 (7)	45101 (24)	39250 (14)	3·4
C(3)	27059 (9)	47303 (28)	29897 (15)	4·5
C(4)	30027 (10)	39175 (32)	24306 (15)	5·0
C(5)	33489 (10)	28889 (31)	28017 (16)	4·8
C(6)	34034 (8)	26788 (26)	37254 (14)	3·8
C(7)	39464 (8)	25919 (23)	72467 (13)	3·2
C(8)	35778 (8)	16798 (28)	75883 (15)	4·1
C(9)	34595 (10)	18502 (33)	84843 (18)	5·2
C(10)	37068 (11)	29320 (34)	90245 (16)	5·4
C(11)	40735 (11)	38464 (32)	86786 (17)	5·3
C(12)	42002 (9)	36958 (27)	77878 (15)	4·2
C(13)	44511 (7)	5612 (24)	61161 (13)	3·2
C(14)	42257 (8)	−8913 (26)	60291 (15)	4·0
C(15)	45194 (12)	−22271 (30)	60703 (18)	5·4
C(16)	50243 (12)	−20872 (38)	62004 (19)	6·3
C(17)	52429 (10)	−6274 (41)	62837 (21)	6·3
C(18)	49607 (8)	7039 (32)	62405 (17)	4·8

Table 2. *Bond lengths* (Å) *and angles* $(°)$ *of non-hydrogen atoms and dimensions about the* S···O *contact*

S(1)–N(1)	1·711 (2)	C(5)–C(6)	1·377 (3)
S(1)–C(1)	1·754 (2)	C(7)–C(8)	1·374 (3)
S(2)–N(1)	1·544 (2)	C(7)–C(12)	1·394 (3)
S(2)–O(1)	1·442 (2)	C(8)–C(9)	1·380 (4)
S(2)–C(7)	1·778 (2)	C(9)–C(10)	1·373 (4)
S(2)–C(13)	1·763 (2)	C(10)–C(11)	1·373 (4)
N(2)–O(21)	1·231 (3)	C(11)–C(12)	1·377 (4)
N(2)–O(22)	1·224 (3)	C(13)–C(14)	1·379 (3)
N(2)–C(2)	1·443 (3)	C(13)–C(18)	1·390 (3)
C(1)–C(2)	1·393 (3)	C(14)–C(15)	1·384 (4)
C(1)–C(6)	1·399 (3)	C(15)–C(16)	1·378 (5)
C(2)–C(3)	1·394 (3)	C(16)–C(17)	1·376 (5)
C(3)–C(4)	1·361 (4)	C(17)–C(18)	1·364 (4)
C(4)–C(5)	1·378 (4)		

N(1)–S(1)–C(1)	99·6 (1)	C(4)–C(5)–C(6)	121·4 (2)
N(1)–S(2)–O(1)	122·7 (1)	C(1)–C(6)–C(5)	121·0 (2)
N(1)–S(2)–C(7)	109·7 (1)	S(2)–C(7)–C(8)	118·7 (2)
N(1)–S(2)–C(13)	102·1 (1)	S(2)–C(7)–C(12)	120·1 (2)
O(1)–S(2)–C(7)	106·8 (1)	C(8)–C(7)–C(12)	121·1 (2)
O(1)–S(2)–C(13)	110·0 (1)	C(7)–C(8)–C(9)	119·4 (2)
C(7)–S(2)–C(13)	104·1 (1)	C(8)–C(9)–C(10)	120·0 (3)
S(1)–N(1)–S(2)	114·1 (1)	C(9)–C(10)–C(11)	120·3 (3)
O(21)–N(2)–O(22)	122·9 (2)	C(10)–C(11)–C(12)	120·8 (3)
O(21)–N(2)–C(2)	117·2 (2)	C(7)–C(12)–C(11)	118·3 (2)
O(22)–N(2)–C(2)	119·8 (2)	S(2)–C(13)–C(14)	121·4 (2)
S(1)–C(1)–C(2)	123·0 (2)	S(2)–C(13)–C(18)	117·0 (2)
S(1)–C(1)–C(6)	120·7 (2)	C(14)–C(13)–C(18)	121·6 (2)
C(2)–C(1)–C(6)	116·3 (2)	C(13)–C(14)–C(15)	118·4 (2)
N(2)–C(2)–C(1)	119·9 (2)	C(14)–C(15)–C(16)	120·0 (3)
N(2)–C(2)–C(3)	117·8 (2)	C(15)–C(16)–C(17)	120·7 (3)
C(1)–C(2)–C(3)	122·3 (2)	C(16)–C(17)–C(18)	120·2 (3)
C(2)–C(3)–C(4)	119·8 (2)	C(13)–C(18)–C(17)	119·0 (3)
C(3)–C(4)–C(5)	119·2 (2)		

S(1)···O(21)	2·532 (2)	C(1)–S(1)···O(21)	75·7 (1)
N(1)–S(1)···O(21)	175·0 (1)	S(1)···O(21)–N(2)	104·1 (2)

Cano & García-Blanco, 1977; Foces-Foces, Cano & García-Blanco, 1979). This is the first case with the imido group attached to S. The bonds about the tetracoordinated sulfur $S^{VI}(2)$ form a distorted tetrahedron as in other sulfoximido groups. The angle N(1)–S(2)–O(1) is 122·7°, which is significantly larger than the tetrahedral angle. For N-sulfoximides the corresponding angles are within the range 118–122°. The torsion angles about the S–N bonds are: C(1)–S(1)–N(1)–S(2) = 123·6 (1)°, O(1)–S(2)–N(1)–S(1) = −64·7 (1)°, C(7)–S(2)–N(1)–S(1) = 61·9 (1)° and C(13)–S(2)–N(1)–S(1) = 171·8 (1)°. The bond angle N(1)–S(2)–C(13) (102·7°), where C(13) is *trans* to S(1) about the N(1)–S(2) bond, is smaller than the other N(1)–S(2)–C(7) angle (109·7°), in which C(7) is *gauche* to S(1). The angle O(1)–S(2)–C(13) (110·0°) is larger than O(1)–S(2)–C(7) (106·8°). The same trends were also found for N-sulfoximides. S(2)–O(1) (1·442 Å) is slightly longer than those of sulfones (1·43 Å). $S^{VI}(2)$–N(1) (1·544 Å) corresponds to the normal S=N double bond (Pauling, 1960). The S=N bonds in N-sulfoximides are found in the range 1·53 to 1·57 Å. Larger values were obtained for dialkylsulfoximide than for diarylsulfoximides. S(1)–N(1) (1·711 Å) is slightly shorter than an S–N single bond. $S^{II}(1)$–C(1) (1·754 Å) of divalent sulfur is also shorter than the other two S^{VI}–C bonds (1·763 and 1·778 Å).

The two phenyl rings of the diarylsulfoximide with a dihedral angle of 78·4 (2)° are both planar with maximum deviation of 0·003 (3) Å. The dihedral angles between the C(7)–S(2)–C(13) plane and the two phenyl rings are 76·5 (2) and 87·8 (2)°, respectively.
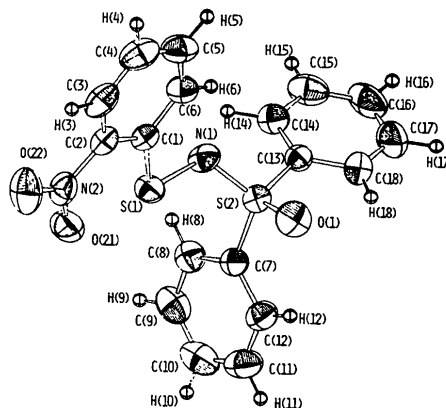

Fig. 1. *ORTEP*II (Johnson, 1971) drawing with atomic numbering. The thermal ellipsoids for non-H atoms are drawn at 50% probability.

The phenyl ring of the *o*-nitrobenzene is more distorted than the other phenyl rings, with deviations within 0·009 (4) Å. The deviations of S(1), N(1), N(2), O(21) and O(22) from this plane are $-0\cdot015$ (3), 0·030 (4), 0·037 (4), 0·006 (4) and 0·107 (4) Å, respectively, which show approximate planarity of the *o*-nitrophenylthio group. Associated with this planarity a very short nonbonding O(21)···S(1) contact of 2·532 (2) Å is observed, while the sum of the van der Waals radii for S and O is 3·25 Å (Pauling, 1960). The dihedral angle between the planes of the nitro group and the benzene ring is 3·0 (2)° and that between the benzene ring and the plane of C(1)–S(1)–N(1) is 1·6 (2)°. O(21)···S(1)–N(1) is approximately linear. This is a favourable situation for sulfur *p*- and *d*-orbital participation in the partial bonding as in thiathiophthenes (dithiolo[2,3-*b*]dithioles) (Hansen & Hordvik, 1973). In the O–S–O dioxy analogue of thiathiophthenes the O–S lengths are 1·85–1·92 Å (Dalseng, Hansen & Hordvik, 1981).

For many nitrobenzene derivatives appreciable twisting about the C—N bond has been found, especially for molecules with steric crowding at the *ortho* position (Herbstein & Kaftory, 1975). But the situation is quite different for 2-nitrophenylthio groups. Short intramolecular contacts between S and O in the 2-nitrophenylthio groups have been found for *N*-(2-nitrophenylthio)phthalimide (2·501 Å; Iwasaki & Masuko, 1986) and other compounds listed in Table 3. The shortest example is *o*-nitrophenylsulfenate (2·44 Å; Hamilton & LaPlaca, 1964), but for the other compounds S···O distances are within the range 2·59–2·70 Å. The S···O lengths change as the electronegativity of the atom (*X*) attached to S. The S···O lengths are 2·619–2·697 Å for *X* = C, 2·589 and 2·636 Å for *X* = S, 2·532 and 2·501 Å for *X* = N and 2·44 Å for *X* = O. The dihedral angles between the benzene ring and the nitro group in these compounds are less than 25°, and the S···O length becomes shorter as the dihedral angle decreases. Other correlations are found among the S···O length, the C(1)–S···*X* angle and the S···O–N(2) angle. The smaller C—S···*X* and the larger S···O—N angles correspond to the shorter S···O lengths.

The N(2)—C(2) length is shorter than the normal N—C single-bond length (1·48 Å) and the N—C lengths for nitrobenzenes where the nitro groups have no conjugation to other groups. The longer C(1)—C(2) and C(1)—C(6) bonds and the shorter C(3)—C(4) and C(5)—C(6) bonds show the contribution of the *ortho* quinonoid character like that of *o*-nitrophenols with an O—H···O intramolecular hydrogen bond (Iwasaki & Kawano, 1977, 1978). For other 2-nitrophenylthio groups a similar situation has been found.

Fig. 2 shows a stereoscopic view of the crystal structure. The intermolecular contacts are of the van der Waals type.

Table 3. *Comparison of 2-nitrophenylthio groups (distances in Å, angles in °)*

	X	S···O	S–C	S–X	C···S–X	C–S···O	C–S–X	S···O–N	ω*
(a)	O	2·44	1·77	1·65	177	77	100	106	
(b)	N	2·501	1·771	1·694	175·7	76·1	99·9	105·2	4·7
(c)	N	2·532	1·754	1·711	175·0	75·7	99·6	104·1	3·0
(d)	S	2·636	1·785	2·043	167·2	76·0	104·3	99·7	18·0
	S	2·589	1·808	2·043	173·5	75·6	104·7	102·7	7·1
(e)	C	2·697	1·752	1·843	172·7	75·1	102·5	98·8	19·2
(f)	C	2·634	1·769	1·808	175·6	74·9	101·9	102·3	3·7
(g)	C	2·619	1·769	1·797	170·0	74·9	103·6	102·9	2·8
(h)	C	2·658	1·761	1·812	175·6	75·2	103·4	100·7	5·8
(i)	C	2·695	1·757	1·813	173·4	74·6	103·7	97·6	23·5
(j)	C	2·643	1·778	1·781	174·2	75·6	102·2	101·7	1·0
(k)	C	2·674	1·766	1·813	173·9	74·4	102·6	98·4	19·4
(l)	C	2·619	1·753	1·816	178·8	76·1	103·8	101·7	4·0
(m)	C	2·657	1·800	1·774	170·6	75·2	102·1	101·4	10·5
(m)	C	2·732	1·729	1·767	165·0	73·3	101·3	97·1	23·9
(n)	C	2·659	1·765	1·801	174·7	74·8	102·4	99·1	17·9
(o)	C	2·694	1·759	1·812	175·5	74·8	104·5	97·3	24·9
(p)	C	2·619	1·757	1·782	179·2	75·7	103·5	101·1	10·2
(p)	C	2·656	1·768	1·777	171·7	74·8	100·8	97·9	23·9
(p)	C	2·637	1·768	1·785	169·8	74·9	102·6	101·7	8·7

References: (*a*) Hamilton & LaPlaca (1964); (*b*) Iwasaki & Masuko (1986); (*c*) present study; (*d*) Ricci & Bernal (1970); (*e*) Rau, Potekhin, Rau, Struchkov, Akhamedova, Zefirov & Sadovaya (1981); (*f*) Zefirov, Kirin, Kozmin, Bodrikov, Potekhin & Kurkutova (1979); (*g*) Zefirov, Kozmin, Kirin, Zhdankin & Caple (1981); (*h*) Sedov, Rau, Rau, Struchkov, Akhamedova, Zefirov & Sadovaya (1980*b*); (*i*) Nersisyan, Yanovsky, Struchkov, Andreeva, Sadovaya & Zefirov (1981); (*j*) Korp, Bernal, Miller, Turley, Williams & Martin (1978); (*k*) Sedov, Rau, Rau, Struchkov, Akhamedova, Zefirov & Sadovaya (1980*a*); (*l*) Zefirov, Kozmin, Zhdankin, Kirin, Bodrikov, Sedov & Rau (1979); (*m*) Barnes, Paton & Nicholla (1982); (*n*) Zefirov, Kirin, Kozmin, Potekhin, Yanovsky & Struchkov (1982); (*o*) Garratt, Przybylska & Cyglar (1983); (*p*) Kucsman, Kapovits, Parkanyi, Argay & Kálmán (1984).
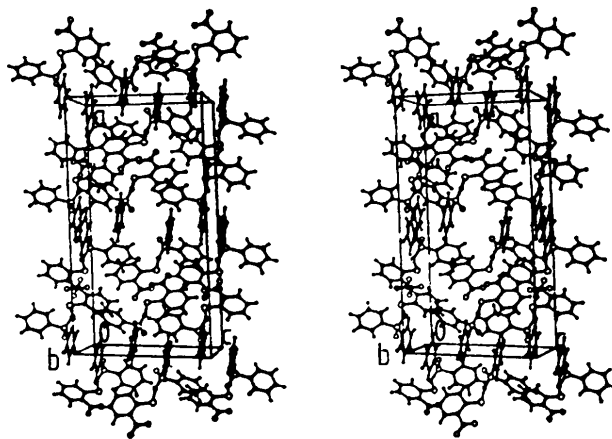* Dihedral angle between the phenyl plane and –C–NO₂ group.

Fig. 2. Stereoscopic view of the crystal structure.

References

AKUTAGAWA, K., FURUKAWA, N. & OAE, S. (1984). *Bull. Chem. Soc. Jpn*, **57**, 518–524.

ANDREETTI, G. D., BOCELLI, G., COGHI, L. & SGARABOTTO, P. (1974). *Cryst. Struct. Commun.* **3**, 765–768.

ANDREETTI, G. D., BOCELLI, G., COGHI, L. & SGARABOTTO, P. (1975). *Cryst. Struct. Commun.* **4**, 393–397.

ANDREETTI, G. D., BOCELLI, G. & SGARABOTTO, P. (1975). *Gazz. Chim. Ital.* **105**, 165–173.

ANDREETTI, G. D., BOCELLI, G. & SGARABOTTO, P. (1977). *Cryst. Struct. Commun.* **6**, 761–765.

BARNES, J. C., PATON, J. D. & NICHOLLA, B. H. (1982). *Acta Cryst.* B**38**, 1525–1529.

CABEZUELO, M. D., FOCES-FOCES, C., CANO, F. H. & GARCÍA-BLANCO, S. (1977). *Acta Cryst.* B**33**, 3911–3913.

DALSENG, M., HANSEN, L. K. & HORDVIK, A. (1981). *Acta Chem. Scand.* A**35**, 645–651.

FOCES-FOCES, C., CANO, F. H. & GARCÍA-BLANCO, S. (1979). *J. Cryst. Mol. Struct.* **9**, 143–151.

GARRATT, D. G., PRZYBYLSKA, M. & CYGLAR, M. (1983). *Can. J. Chem.* **61**, 1176–1184.

HAMILTON, W. C. & LAPLACA, S. J. (1964). *J. Am. Chem. Soc.* **86**, 2289–2290.

HANSEN, L. K. & HORDVIK, A. (1973). *Acta Chem. Scand.* **27**, 411–420.

HERBSTEIN, F. H. & KAFTORY, M. (1975). *Acta Cryst.* B**31**, 68–75.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

IWASAKI, F. & KAWANO, Y. (1977). *Acta Cryst.* B**33**, 2455–2459.

IWASAKI, F. & KAWANO, Y. (1978). *Acta Cryst.* B**34**, 1286–1290.

IWASAKI, F. & MASUKO, Y. (1986). *Acta Cryst.* C**42**, 124–127.

JOHNSON, C. K. (1971). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.

KORP, J. D., BERNAL, I., MILLER, R. F., TURLEY, J. C., WILLIAMS, L. & MARTIN, G. E. (1978). *J. Cryst. Mol. Struct.* **8**, 127–140.

KUCSMAN, A., KAPOVITS, I., PARKANYI, L., ARGAY, GY. & KÁLMÁN, A. (1984). *J. Mol. Struct.* **125**, 331–347.

MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

NERSISYAN, A. M., YANOVSKY, A. I., STRUCHKOV, YU. T., ANDREEVA, L. A., SADOVAYA, N. K. & ZEFIROV, N. S. (1981). *Cryst. Struct. Commun.* **10**, 1457–1460.

PAULING, L. (1960). *The Nature of the Chemical Bond*, 3rd ed. Ithaca: Cornell Univ. Press.

RAU, T. F., POTEKHIN, K. A., RAU, V. G., STRUCHKOV, YU. T., AKHAMEDOVA, R. S., ZEFIROV, N. S. & SADOVAYA, N. K. (1981). *Cryst. Struct. Commun.* **10**, 583–586.

RICCI, J. S. & BERNAL, I. (1970). *J. Chem. Soc. B*, pp. 806–811.

SAKURAI, T. & KOBAYASHI, K. (1979). *Rikagaku Kenkyusho Hokoku*, **55**, 69–77.

SEDOV, B. B., RAU, T. F., RAU, V. G., STRUCHKOV, YU. T., AKHAMEDOVA, R. S., ZEFIROV, N. S. & SADOVAYA, N. K. (1980a). *Cryst. Struct. Commun.* **9**, 633–637.

SEDOV, B. B., RAU, T. F., RAU, V. G., STRUCHKOV, YU. T., AKHAMEDOVA, R. S., ZEFIROV, N. S. & SADOVAYA, N. K. (1980b). *Cryst. Struct. Commun.* **9**, 639–642.

ZEFIROV, N. S., KIRIN, V. N., KOZMIN, A. S., BODRIKOV, I. V., POTEKHIN, K. A. & KURKUTOVA, E. N. (1979). *Tetrahedron Lett.* pp. 1547–1550.

ZEFIROV, N. S., KIRIN, V. N., KOZMIN, A. S., POTEKHIN, K. A., YANOVSKY, A. I. & STRUCHKOV, YU. T. (1982). *Cryst. Struct. Commun.* **11**, 721–726.

ZEFIROV, N. S., KOZMIN, A. S., KIRIN, V. N., ZHDANKIN, V. V. & CAPLE, R. (1981). *J. Org. Chem.* **46**, 5264–5275.

ZEFIROV, N. S., KOZMIN, A. S., ZHDANKIN, V. V., KIRIN, V. N., BODRIKOV, I. V., SEDOV, B. B. & RAU, V. G. (1979). *Tetrahedron Lett.* pp. 3533–3536.

Structure of *N*-(2-Nitrophenylthio)phthalimide. Intramolecular S···O Interaction

BY FUJIKO IWASAKI* AND YOSHIAKI MASUKO

Department of Materials Science, University of Electro-Communications, Chofu-shi, Tokyo 182, Japan

Abstract. $C_{14}H_8N_2O_4S$, $M_r = 300\cdot29$, monoclinic, $P2_1/c$, $a = 12\cdot037$ (1), $b = 13\cdot710$ (2), $c = 7\cdot858$ (1) Å, $\beta = 99\cdot94$ (1)°, $V = 1277\cdot4$ (2) Å^3, $Z = 4$, $D_x = 1\cdot561$ Mg m^{-3}, Mo $K\alpha_1$, $\lambda = 0\cdot70926$ Å, $\mu = 0\cdot258$ mm^{-1}, $F(000) = 616$, $T = 293$ K, $R = 0\cdot041$ for 2405 observed reflexions. The molecule is composed of two planar sections, the thiophthalimido and 2-nitrophenylthio groups, which form a dihedral angle of $83\cdot3$ (3)°, and has an approximate mirror plane coinciding with the 2-nitrophenyl plane and bisecting the phthalimido group. A short intramolecular contact [$2\cdot501$ (2) Å] is found between the O atom of the nitro group and S. The angle O···S—N is $175\cdot7$ (1)°. The calculation of the total energy by the CNDO/2 method as a function of torsion angle (φ) of the nitro group shows a minimum value at $\varphi = -10°$, which is consistent with the observed angle of $-3\cdot8$ (3)°.

Introduction. The title compound was synthesized by Oae & Kodera (1983). For the 2-nitrophenylthio group a short intramolecular S···O interaction is expected. An X-ray analysis was undertaken to investigate the structural features of this compound.

* To whom all correspondence should be addressed.