DOMENICANO, A. & MURRAY-RUST, P. (1979). *Tetrahedron Lett.* **24**, 2283–2286.

DOMENICANO, A., SCHULTZ, G., HARGITTAI, I., COLAPIETRO, M., PORTALONE, G., GEORGE, P. & BOCK, C. W. (1990). *Struct. Chem.* **1**, 107–122.

DOMENICANO, A., VACIAGO, A. & COULSON, C. A. (1975). *Acta Cryst.* B**31**, 221–234.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

KRYGOWSKY, T. M. (1990). *Prog. Phys. Org. Chem.* **17**, 239–291.

LANGSETH, A. & STOICHEFF, B. P. (1956). *Can. J. Phys.* **34**, 350–353.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

MAK, T. C. W. & TROTTER, J. (1965). *Acta Cryst.* **18**, 68–74.

MAURIN, J. & KRYGOWSKY, T. M. (1988). *J. Mol. Struct.* **172**, 413–421.

MAVERICK, E. F. & TRUEBLOOD, K. N. (1988). *THMA11*. Program for thermal motion analysis. Univ. of California, Los Angeles, USA.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

NORRESTAM, R. & SCHEPPER, L. (1981). *Acta Chem. Scand. Ser. A*, **35**, 91–103.

NUDELMAN, N. S., SOCOLOVSKY, S. E. & DE WAISBAUM, R. G. (1982). *An. Quim.* **78C**, 145–148.

NYBURG, S. C., FAERMAN, C. H., PRASAD, L., PALLEROS, D. R. & NUDELMAN, N. S. (1987). *Acta Cryst.* C**43**, 686–689.

PANUNTO, T. W., URBÁNCZYK-LIPOWSKA, Z., JOHNSON, R. & ETTER, M. C. (1987). *J. Am. Chem. Soc.* **109**, 7786–7797.

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

PRASAD, L., GABE, E. J. & LEPAGE, Y. (1982). *Acta Cryst.* B**38**, 674–675.

PUNTE, G. & RIVERO, B. E. (1991). *Acta Cryst.* C**47**. In the press.

PUNTE, G., RIVERO, B. E., SOCOLOVSKY, S. & NUDELMAN, N. S. (1987). *Acta Cryst.* A**43**, C-169.

PUNTE, G., RIVERO, B. E., SOCOLOVSKY, S. & NUDELMAN, N. S. (1989). *Acta Cryst.* C**45**, 1952–1957.

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

TRUEBLOOD, K. N., GOLDISH., E. & DONOHUE, J. (1961). *Acta Cryst.* **14**, 1009–1017.

Acta Cryst. (1991). C**47**, 1227–1230

Structures of an Ylide 4,4,5,5,6,6-Hexafluoro-2-(phenyliodonio)dithianide 1,1,3,3-Tetraoxide (I) and its Complex with Dimethyl Sulfoxide (II)

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Abstract. (I), $C_{10}H_5F_6IO_4S_2$, $M_r = 494{\cdot}2$, monoclinic, $P2_1/n$, $a = 7{\cdot}387\ (3)$, $b = 20{\cdot}403\ (8)$, $c = 10{\cdot}206\ (3)$ Å, $\beta = 99{\cdot}28\ (3)^\circ$, $V = 1518$ Å^3, $Z = 4$, $D_x = 2{\cdot}162$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 24{\cdot}27$ cm^{-1}, $F(000) = 944$, room temperature, final $R = 0{\cdot}073$ for 1757 observed unique reflections. (II), $C_{10}H_5F_6IO_4S_2.C_2H_6OS$, $M_r = 572{\cdot}3$, monoclinic, $P2_1/n$, $a = 8{\cdot}733\ (3)$, $b = 10{\cdot}239\ (2)$, $c = 21{\cdot}101\ (1)$ Å, $\beta = 94{\cdot}47^\circ$, $V = 1881$ Å^3, $Z = 4$, $D_x = 2{\cdot}021$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 20{\cdot}79$ cm^{-1}, $F(000) = 1112$, room temperature, final $R = 0{\cdot}040$ for 2158 observed unique reflections. The crystal structures show that the I atom in both compounds bonds to the methyl C atom of hexafluorodithianide tetraoxide to form an iodonium ylide containing a strong polar bond between the carbanion and the I atom. In (II), the I atom also interacts with the O atom of Me_2SO through charge transfer. The sulfonyl groups with electron deficiency accept electrons from the carbanion by resonance. The resonance delocalizes the negative charge of the carbanion, thereby stabilizing the carbanion and leading to the stable crystallization of the ylide and its complex at room temperature.

Introduction. Phenyliodonium disulfonylmethylides (1), one of the classes of carbene precursors, have aroused great interest because they can react with many kinds of alkenes under a variety of conditions to yield expected products. However, these ylides are difficult to crystallize because of their decomposition at room temperature (Hatjiarapoglou & Varvoglis, 1988), which may be attributed to the instability of the bonding between iodonium and methyl C atoms. Up to now there have been few crystal structure reports about iodonium ylides. As the introduction

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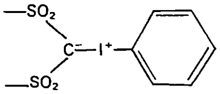
of some electron-attracting groups into the part of disulfonylmethyl group probably improves the stability of the $C^-—I^+$ bond, we prepared 4,4,5,5,6,6-hexafluoro-1-(phenylidonio)dithianide 1,1,3,3-tetraoxide (I) and phenyliodonium bis(perfluoroallylsulfonyl)methylide, *etc.* (Chen, 1991). As expected, these products could be recrystallized and analyzed by X-ray diffraction at room temperature. NMR studies on the product obtained from the reaction of the title ylide with Me_2SO revealed that the phenyliodonium group was not detached from the ylide and Me_2SO existed in the product simultaneously. For further identification of the structures of the iodonium ylide and its product, the structural determinations of the title ylide compounds were undertaken.



(1)

Experimental. For (I), colourless crystal 0·1 × 0·2 × 0·1 mm, D_m not measured, Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo *Kα* radiation, $\omega/2\theta$ scanning technique, room temperature. Cell parameters defined by 25 reflections with $12 < \theta < 17°$. 3500 unique reflections ($R_{\rm int} = 0·006$) measured with $2\theta_{\max} = 50°$, $0 \leq h \leq 8$, $0 \leq k \leq 24$, $-12 \leq l \leq 12$. Three standard reflections monitored every 1·5 h, no significant variation found. Lp and empirical absorption correction with factors 0·71 to 0·99. Structure solved by direct methods. Full-matrix least-squares refinement on *F* with anisotropic temperature factors for non-H atoms (244 variables), 1757 observed reflections [$F^2 > 3\sigma(F^2)$] with unit weights used in refinement, $R = 0·073$, $wR = 0·094$, $S = 2·45$, $\rho_{\max} = 1·77$ e Å^{-3} (0·937 Å from the I atom), $(\Delta/\sigma)_{\max} = 0·01$. For (II), colourless crystal 0·2 × 0·2 × 0·3 mm, D_m not measured, CAD-4 diffractometer, graphite-monochromated Mo *Kα* radiation, $\omega/2\theta$ scanning technique. 25 reflections with $14 < \theta < 16°$ to determine the cell parameters. Data collected with $2\theta_{\max} = 50°$, $0 \leq h \leq 10$, $0 \leq k \leq 12$, $-25 \leq l \leq 25$, 3765 unique reflections measured ($R_{\rm int} = 0·018$). Three standard reflections checked every hour, no obvious changes observed, Lp and empirical absorption correction with factors 0·92 to 1·00. Structure determined *via* direct methods. All positional and anisotropic thermal parameters (244 variables) refined on *F* for 2158 observed reflections with $F^2 > 3\sigma(F^2)$ by full-matrix least squares, $R = 0·040$, $wR = 0·048$, $S = 2·79$, $\rho_{\max} = 1·382$ e Å^{-3} (1·159 Å from the I atom), $(\Delta/\sigma)_{\max} = 0·01$. All computations performed on a MicroVAX II computer with programs *SDP-Plus* (B. A. Frenz & Associates Inc., 1984), *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and *ORTEP*II (Johnson, 1976). Atomic scattering factors taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Table 1. *Positional and thermal parameters with e.s.d.'s for* (I)

$$B_{\rm eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	*y*	*z*	$B_{\rm eq}$(Å^2)
I	0·6345 (2)	0·24466 (6)	0·4560 (1)	3·56 (2)
S(1)	0·3405 (5)	0·3464 (2)	0·3009 (4)	3·09 (8)
S(2)	0·6670 (6)	0·3228 (3)	0·1829 (4)	3·7 (1)
F(1)	0·283 (2)	0·4717 (7)	0·319 (1)	7·0 (4)
F(2)	0·525 (2)	0·4342 (6)	0·447 (1)	5·3 (3)
F(3)	0·449 (2)	0·4582 (7)	0·107 (1)	6·7 (3)
F(4)	0·609 (2)	0·5136 (7)	0·261 (2)	8·0 (4)
F(5)	0·804 (2)	0·4341 (7)	0·120 (1)	7·2 (3)
F(6)	0·837 (1)	0·4150 (6)	0·331 (1)	5·1 (3)
O(1)	0·230 (2)	0·3474 (7)	0·176 (1)	4·0 (3)
O(2)	0·266 (2)	0·3294 (8)	0·420 (1)	5·3 (3)
O(3)	0·559 (2)	0·3225 (8)	0·052 (1)	4·5 (3)
O(4)	0·836 (2)	0·2892 (8)	0·210 (2)	5·6 (4)
C(1)	0·536 (2)	0·3029 (9)	0·300 (2)	3·4 (4)
C(2)	0·424 (2)	0·430 (1)	0·328 (2)	4·0 (4)
C(3)	0·547 (3)	0·454 (1)	0·226 (2)	4·2 (4)
C(4)	0·721 (3)	0·411 (1)	0·221 (2)	4·9 (4)
C(5)	0·766 (2)	0·3110 (9)	0·594 (1)	2·6 (3)
C(6)	0·935 (2)	0·335 (1)	0·582 (2)	4·1 (4)
C(7)	1·021 (3)	0·378 (1)	0·682 (2)	5·0 (5)
C(8)	0·936 (4)	0·396 (1)	0·782 (2)	6·5 (6)
C(9)	0·766 (3)	0·372 (1)	0·794 (2)	4·6 (4)
C(10)	0·678 (3)	0·329 (1)	0·705 (2)	4·9 (4)

Table 2. *Positional and thermal parameters with e.s.d.'s for* (II)

$$B_{\rm eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	*y*	*z*	$B_{\rm eq}$(Å^2)
I	0·69387 (7)	0·19782 (6)	0·95651 (3)	3·34 (1)
S(1)	0·5043 (3)	−0·0275 (2)	0·8743 (1)	3·52 (5)
S(2)	0·3231 (3)	0·1543 (2)	0·9408 (1)	3·48 (5)
S(3)	1·0157 (3)	0·3548 (3)	1·0541 (1)	4·50 (6)
F(1)	0·3904 (8)	−0·0582 (6)	0·7575 (3)	5·9 (2)
F(2)	0·4958 (7)	0·1296 (6)	0·7772 (3)	4·7 (1)
F(3)	0·1985 (7)	0·1541 (6)	0·7529 (3)	5·9 (1)
F(4)	0·1485 (7)	0·0028 (6)	0·8200 (3)	5·5 (1)
F(5)	0·0932 (7)	0·2366 (7)	0·8648 (3)	6·2 (2)
F(6)	0·3169 (7)	0·3086 (5)	0·8427 (3)	4·8 (1)
O(1)	0·6570 (8)	−0·0508 (7)	0·8588 (3)	4·9 (2)
O(2)	0·4075 (9)	−0·1334 (6)	0·8908 (3)	4·9 (2)
O(3)	0·2225 (8)	0·0551 (7)	0·9602 (3)	4·8 (2)
O(4)	0·3403 (8)	0·2730 (7)	0·9771 (3)	4·9 (2)
O(5)	0·9342 (8)	0·3323 (7)	0·9902 (3)	4·7 (2)
C(1)	0·497 (1)	0·0994 (8)	0·9249 (4)	2·9 (2)
C(2)	0·406 (1)	0·0387 (9)	0·8002 (4)	3·7 (2)
C(3)	0·247 (1)	0·099 (1)	0·8090 (4)	4·1 (2)
C(4)	0·239 (1)	0·203 (1)	0·8604 (5)	4·1 (2)
C(5)	0·678 (1)	0·3556 (9)	0·8920 (4)	3·2 (2)
C(6)	0·731 (1)	0·336 (1)	0·8321 (4)	4·5 (2)
C(7)	0·715 (1)	0·442 (1)	0·7894 (6)	7·2 (3)
C(8)	0·651 (1)	0·555 (1)	0·8072 (6)	7·6 (3)
C(9)	0·602 (1)	0·574 (1)	0·8683 (7)	6·7 (3)
C(10)	0·615 (1)	0·470 (1)	0·9127 (6)	4·7 (2)
C(11)	1·023 (1)	0·201 (1)	1·0952 (6)	6·8 (3)
C(12)	0·878 (2)	0·433 (1)	1·0986 (6)	7·8 (4)

Table 3. *Bond lengths* (Å) *and bond angles* (°) *for* (I)

I—C(1)	2·029 (7)	F(3)—C(3)	1·316 (9)
I—C(5)	2·089 (7)	F(4)—C(3)	1·316 (9)
S(1)—O(1)	1·392 (5)	F(5)—C(4)	1·388 (9)
S(1)—O(2)	1·439 (6)	F(6)—C(4)	1·28 (1)
S(1)—C(1)	1·702 (7)	C(2)—C(3)	1·56 (1)
S(1)—C(2)	1·851 (9)	C(3)—C(4)	1·59 (1)
S(2)—O(3)	1·436 (5)	C(5)—C(6)	1·39 (1)
S(2)—O(4)	1·411 (7)	C(5)—C(10)	1·44 (2)
S(2)—C(1)	1·691 (7)	C(6)—C(7)	1·42 (1)
S(2)—C(4)	1·86 (2)	C(7)—C(8)	1·38 (2)
F(1)—C(2)	1·325 (9)	C(8)—C(9)	1·41 (2)
F(2)—C(2)	1·34 (1)	C(9)—C(10)	1·34 (1)

C(1)—I—C(5)	102·4 (3)	F(1)—C(2)—C(3)	107·4 (7)
O(1)—S(1)—O(2)	121·4 (4)	F(2)—C(2)—C(3)	108·0 (7)
O(1)—S(1)—C(1)	112·6 (3)	F(3)—C(3)—F(4)	107·5 (8)
O(1)—S(1)—C(2)	105·2 (4)	F(3)—C(3)—C(2)	110·2 (7)
O(2)—S(1)—C(1)	108·7 (3)	F(3)—C(3)—C(4)	109·3 (8)
O(2)—S(1)—C(2)	105·2 (4)	F(4)—C(3)—C(2)	109·3 (8)
C(1)—S(1)—C(2)	101·7 (4)	F(4)—C(3)—C(4)	106·0 (7)
O(3)—S(2)—O(4)	121·1 (5)	S(2)—C(4)—F(5)	106·0 (7)
O(3)—S(2)—C(1)	111·5 (3)	S(2)—C(4)—F(6)	110·6 (7)
O(3)—S(2)—C(4)	105·6 (4)	S(2)—C(4)—C(3)	113·7 (6)
O(4)—S(2)—C(1)	109·4 (5)	F(5)—C(4)—F(6)	108·9 (8)
O(4)—S(2)—C(4)	105·8 (4)	R(5)—C(4)—C(3)	106·9 (8)
C(1)—S(2)—C(4)	101·4 (4)	F(6)—C(4)—C(3)	110·5 (8)
I—C(1)—S(1)	119·7 (4)	C(6)—C(5)—C(10)	123·7 (7)
I—C(1)—S(2)	121·7 (4)	C(5)—C(6)—C(7)	116·3 (7)
S(1)—C(1)—S(2)	117·9 (5)	C(6)—C(7)—C(8)	120·3 (9)
S(1)—C(2)—F(1)	109·0 (6)	C(7)—C(8)—C(9)	121 (1)
S(1)—C(2)—F(2)	108·5 (7)	C(8)—C(9)—C(10)	121·6 (8)
S(1)—C(2)—C(3)	113·7 (6)	C(5)—C(10)—C(9)	116·9 (7)
F(1)—C(2)—F(2)	110·2 (8)		

Table 4. *Bond lengths* (Å) *and bond angles* (°) *for* (II)

I—O(5)	2·564 (4)	F(1)—C(2)	1·340 (6)
I—C(1)	2·058 (5)	F(2)—C(2)	1·336 (7)
I—C(5)	2·110 (6)	F(3)—C(3)	1·350 (7)
S(1)—O(1)	1·418 (5)	F(4)—C(3)	1·337 (7)
S(1)—O(2)	1·434 (4)	F(5)—C(4)	1·330 (6)
S(1)—C(1)	1·686 (5)	F(6)—C(4)	1·350 (7)
S(1)—C(2)	1·853 (6)	C(2)—C(3)	1·350 (7)
S(2)—O(3)	1·424 (4)	C(3)—C(4)	1·524 (9)
S(2)—O(4)	1·439 (4)	C(5)—C(6)	1·392 (8)
S(2)—C(1)	1·678 (6)	C(5)—C(10)	1·378 (8)
S(2)—C(4)	1·862 (6)	C(6)—C(7)	1·41 (2)
S(3)—O(5)	1·493 (4)	C(7)—C(8)	1·35 (1)
S(3)—C(11)	1·794 (7)	C(8)—C(9)	1·40 (1)
S(3)—C(12)	1·768 (8)	C(9)—C(10)	1·42 (1)

O(5)—I—C(1)	176·1 (2)	S(1)—C(2)—C(3)	114·1 (4)
O(5)—I—C(5)	77·1 (2)	F(1)—C(2)—F(2)	107·5 (5)
C(1)—I—C(5)	99·0 (2)	F(1)—C(2)—C(3)	109·5 (5)
O(1)—S(1)—O(2)	120·8 (3)	F(2)—C(2)—C(3)	109·1 (5)
O(1)—S(1)—C(1)	111·0 (3)	F(3)—C(3)—F(4)	107·4 (4)
O(1)—S(1)—C(2)	104·5 (3)	F(3)—C(3)—C(2)	106·5 (6)
O(2)—S(1)—C(1)	112·1 (3)	F(3)—C(3)—C(4)	107·6 (6)
O(2)—S(1)—C(2)	103·7 (3)	F(4)—C(3)—C(2)	108·9 (6)
C(1)—S(1)—C(2)	102·4 (3)	F(4)—C(3)—C(4)	109·0 (5)
O(3)—S(2)—O(4)	119·4 (3)	C(2)—C(3)—C(4)	117·1 (5)
O(3)—S(2)—C(1)	114·0 (3)	S(2)—C(4)—F(5)	108·5 (4)
O(3)—S(2)—C(4)	104·0 (3)	S(2)—C(4)—F(6)	107·1 (4)
O(4)—S(2)—C(1)	109·3 (3)	S(2)—C(4)—C(3)	115·3 (4)
O(4)—S(2)—C(4)	106·2 (3)	F(5)—C(4)—F(6)	108·3 (5)
C(1)—S(2)—C(4)	102·0 (3)	F(5)—C(4)—C(3)	109·2 (5)
O(5)—S(3)—C(11)	107·3 (3)	F(6)—C(4)—C(3)	108·2 (5)
O(5)—S(3)—C(12)	104·7 (3)	C(6)—C(5)—C(10)	125·2 (6)
C(11)—S(3)—C(12)	97·8 (5)	C(5)—C(6)—C(7)	116·8 (8)
I—C(1)—S(1)	120·7 (3)	C(6)—C(7)—C(8)	7120·2 (8)
I—C(1)—S(2)	121·1 (3)	C(7)—C(8)—C(9)	122·4 (8)
S(1)—C(1)—S(2)	117·6 (3)	C(8)—C(9)—C(10)	119·2 (8)
S(1)—C(2)—F(1)	108·3 (4)	C(5)—C(10)—C(9)	116·2 (7)
S(1)—C(2)—F(2)	108·3 (4)		

Discussion. The positional and thermal parameters for (I) and (II) are listed in Tables 1* and 2, respectively. Bond distances and angles for (I) and (II) are given in Tables 3 and 4, respectively. The molecular structures of (I) and (II) are shown in Figs. 1 and 2. In the overall structure of (I), a phenyliodonium group is linked to a hexafluorodithianide tetraoxide group through the C(1)—I bond. The average bond lengths C_{sp^3}—C_{sp^3} = 1·57, F—C_{sp^3} = 1·33, C_{Ph}—C_{Ph} = 1·40 Å agree with the normal values of corresponding bonds. The C(1) atom, exhibiting a carbanion property with sp^2 hybridization, links directly to a heteroatom, the I atom. This structural characteristic is consistent with that in phosphorus ylides in which the C—P bond shows considerable polarity (Schmidbaur, 1973; Johnson, 1966). Consequently, it can be inferred that the I atom carries a high degree of positive charge and the p–$d\pi$ overlap between the

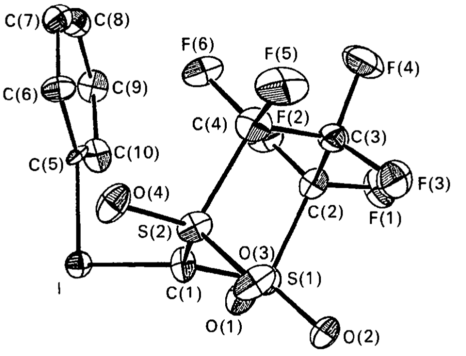


Fig. 1. A view of (I) with atom-numbering scheme.

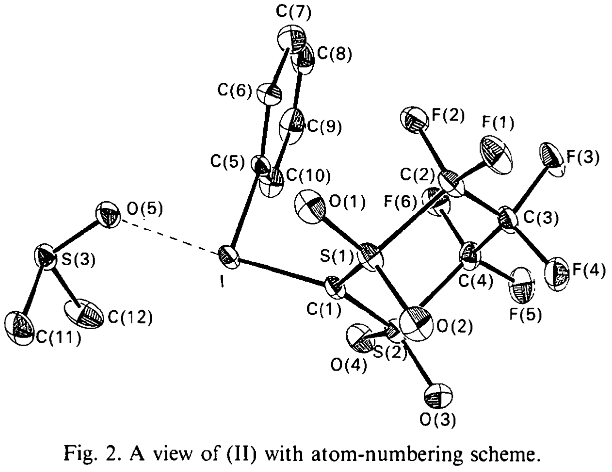


Fig. 2. A view of (II) with atom-numbering scheme.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53581 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

filled 2*p* orbital of the C(1) atom and the vacant *d* orbital of the I atom probably occurs similarly to that in phosphorus ylides (Johnson, 1966). The evidence for the assumption of orbital overlap has been provided from the fact that the C(1)—I bond is shorter than C(5)—I. The S(1)—C(1) and S(2)—C(1) bonds are obviously shorter than S(1)—C(2) and S(2)—C(4). The bond retrenchment reveals that the O═S—C$^-$ structure probably changes into its resonance structures O(1)$^-$—S(1)═C(1), O(2)$^-$—S(1)═C(1), O(3)$^-$—S(2)═C(1) and O(4)$^-$—S(2)═C(1) because the bonds C(1)—S(1) and C(1)—S(2) represent a double bond in four resonance structures. Clearly, the resonances delocalize the negative charge of the carbanion C(1), thereby stabilizing the carbanion structure and leading to an easy crystallization of the title compounds at room temperature. However, some phenyliodonium ylides containing sulfonyl groups without CF_2 groups are unstable at room temperature (Hatjiarapoglou & Varvoglis, 1988). Based on the above fact, it can be considered that the strong electron attraction of adjacent CF_2 groups aggravates the electron deficiency of the SO_2 group and therefore the SO_2 group tends to accept an electron from the carbanion by resonance. A similar situation has been found in some phosphorus ylides with a carbonyl group (Fliszar, Hudson & Salvadori, 1963; Johnson, 1966). Structure (II) consists of a title ylide molecule and an Me_2SO molecule. The configuration of the ylide in the crystal is generally consistent with that of (I) but the precision of structural parameters of the former seems better than that of the latter due to the crystal quality. The shortest contact between the ylide and Me_2SO is I—O(5) at 2·564 (4) Å. This distance implies that the interaction between the ylide and Me_2SO is different from a van der Waals interaction by which host molecules usually interact with solvent molecules in most crystals. It can be inferred that the I atom can either as an electron donor coordinate with the C(1) atom or as an acceptor receive the lone-pair electrons from O(5) atoms to form a charge-transfer complex (Cotton, 1972; Foster, 1973). The C(1)—I···O(5) bond angle [176·1 (2)°] shows a linear linkage among the three atoms. This type of charge-transfer bonding behaviour is similar to that in the complex of 1,4-dioxane with iodine monochloride in which the O···I distance = 2·57 Å and the O···I—Cl system is linear (Hassel & Hvoslef, 1956).

References

B. A. Frenz & Associates Inc. (1984). *SDP-Plus Structure Determination Package*. College Station, Texas, USA.

Chen, Q.-Y. (1991). In preparation.

Cotton, F. A. (1972). *Advanced Inorganic Chemistry*, pp. 563–564. New York: Interscience.

Fliszar, S., Hudson, R. F. & Salvadori, G. (1963). *Helv. Chim. Acta*, **46**, 1580–1584.

Foster, R. (1973). *Molecular Complexes*, pp. 164–166. London: Elck Science.

Hassel, O. & Hvoslef, J. (1956). *Acta Chem. Scand.* **10**, 138–141.

Hatjiarapoglou, L. & Varvoglis, A. (1988). *J. Chem. Soc. Perkin Trans.* 1, pp. 2839–2846.

Johnson, A. W. (1966). *Ylid Chemistry*, pp. 1–4, 65–67, 80–82. New York: Academic Press.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Schmidbaur, H. (1973). *Chem. Ber.* **106**, 1251–1255.

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Structure of a Chiral Tricyclic Phosphonamide

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Abstract. 2-[1-(1,3-Dithiolan-2-ylideneamino)-3-butenyl]-2,3,3a,4,5,6,7,7a-octahydro-1,3-dimethyl-1*H*-1,3,2-benzodiazaphosphole 2-oxide, $C_{15}H_{26}N_3OPS_2$, $M_r = 359·48$, trigonal, $P3_121$, $a = b = 9·778\ (7)$, $c = 34·214\ (1)$ Å, $V = 2832·9$ Å^3, $Z = 6$, $D_x = 1·264$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1·54178$ Å, $\mu(\mathrm{Cu}\ K\alpha) = 3·37$ mm^{-1}, $F(000) = 3552$, $T = 190$ K, $R = 0·046$ for 1991 observed reflections. The title compound contains two heterocyclic rings, a 1,3-diazaphosphole 2-oxide and a 1,3-dithiolanylideneaminobutene bonded to the P atom which is tetrahedrally coordinated. Both five-membered rings are in the half-chair conformation. The diaza ring is *trans*-fused to a cyclohexane which is in the chair conformation. The

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