

Table 3. Power series expansion of $\nu = f(T)$

$f(T) = \sum_{i=-1}^z a_i T^i$; ν_b = bleaching out frequency; T_b = bleaching out temperature; the temperature range in which the least-squares adjustment is valid is for ν_1, ν_4, ν_5 : 77–310.4 K, for ν_2, ν_3, ν_6 : 77–330.1 K; z = number of data; σ = standard deviation.

Sign	$\nu(^{35}\text{Cl})^*$ (MHz)	T (K)	$\nu(^{35}\text{Cl})$ (MHz)	T (K)	$\nu_b(^{35}\text{Cl})^\dagger$ (MHz)	T_b (K)	z	σ (kHz)	a_{-1} (MHz K)	a_0 (MHz)	$a_1 \times 10^{-3}$ (MHz K ⁻¹)	$a_2 \times 10^{-6}$ (MHz K ⁻²)
ν_1	38.786	(77)	37.681	(300.3)	37.623	(310.4)	10	3.6	-1.394	39.076	-3.170	-4.848
ν_2	38.626	(77)	37.745	(300.3)	—	—	12	2.2	4.246	38.718	-1.415	-6.259
ν_3	38.285	(77)	37.247	(300.3)	—	—	12	2.8	2.484	38.522	-3.199	-3.605
ν_4	38.196	(77)	36.532	(300.3)	36.440	(310.4)	13	3.2	-20.442	39.053	-7.523	-2.168
ν_5	38.116	(77)	36.761	(300.3)	36.685	(310.4)	13	2.5	-13.600	38.749	-5.739	-2.448
ν_6	38.058	(77)	36.888	(300.3)	—	—	15	4.7	3.593	38.341	-4.030	-2.892

* The signal to noise ratio (lock-in technique, recorder and time constant 10 s) is 20 to 25.

† The signal to noise ratio is 2–3 near the bleaching out temperature.

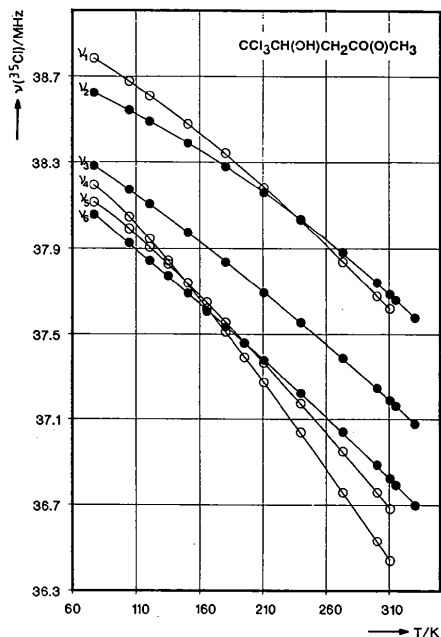


Fig. 2. ³⁵Cl nuclear quadrupole resonance frequencies as a function of temperature.

electric field gradient exists; this in turn leads to six different resonance frequencies at constant temperature. We find two groups of frequencies: the signals of one group bleach out at 310.4 K (above this temperature they become unobservable owing to line broadening) and the signals of the other group are observable nearly up to the melting point of the title compound (331.8 K; Ross, 1936). According to the NQR results, there is no phase transition between 77 K and the melting point.

One of us (RB) is indebted to the Physics Department, Faculty of Science, University of Istanbul, for permitting leave of absence.

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Acta Cryst. (1991). C47, 2402–2406

Absolute Structures of (–)-(S_S,S_C)-1-Phenylethyl 2-Pyridyl Sulfoxide and (–)-(R)-1-Methyl-2-(1-phenylethyl)pyridinium Perchlorate

BY FUJIKO IWASAKI* AND NORIMASA YAMAZAKI

Department of Applied Physics and Chemistry, The University of Electro-Communications, Chofu-shi, Tokyo 182, Japan

(Received 28 December 1990; accepted 22 May 1991)

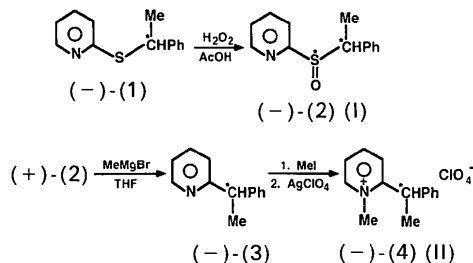
Abstract. (I) C₁₃H₁₃NOS, $M_r = 231.31$, monoclinic, $P2_1$, $a = 14.0654$ (8), $b = 7.7361$ (6), $c = 5.7751$ (3) Å,

$\beta = 94.892$ (6)°, $V = 626.11$ (5) Å³, $Z = 2$, $D_x = 1.227$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.54184$ Å, $\mu = 2.073$ mm⁻¹, $F(000) = 244$, $T = 295$ K, $R = 0.029$ for 2064 observed reflections. (II) C₁₄H₁₆N⁺.ClO₄⁻, M_r ,

* To whom all correspondence should be addressed.

= 297.74, monoclinic, $P2_1$, $a = 12.587(1)$, $b = 7.716(1)$, $c = 15.886(1)$ Å, $\beta = 108.99(1)^\circ$, $V = 1459.0(3)$ Å³, $Z = 4$, $D_x = 1.356$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 2.455$ mm⁻¹, $F(000) = 624$, $T = 295$ K, $R = 0.062$ for 4399 observed reflections. In (I) the S=O bond is nearly in the same plane of the pyridine ring and the O atom is located far from the non-bonding electron pair of the N atom. The absolute configurations of these compounds were determined to be S_S, S_C and R for (I) and (II), respectively. The configuration of 1-phenylethyl group is retained completely during the ligand-coupling reaction of (I) with Grignard reagents.

Introduction. 1-Phenylethyl 2-pyridyl sulfide (I) was separated into two enantiomers from its racemate by mechanical resolution (Furukawa, Kawai, Oae & Iwasaki, 1984). The oxidation of (-)-(1) resulted in the sulfoxide (-)-(2) and the reaction of (-)-(2) with methylmagnesium bromide yielded (+)-2-(1-phenylethyl)pyridine, (+)-(3). The other enantiomer of (3), (-)-(3), was obtained from (+)-(1) via (+)-(2). (-)-(3) was converted into (-)-1-methyl-2-(1-phenylethyl)pyridinium perchlorate, (-)-(4), to be subjected to the determination of the absolute configuration by the X-ray method (Oae, Kawai & Furukawa, 1984; Oae, Kawai, Furukawa & Iwasaki, 1987). The absolute configurations and the molecular conformations of (-)-(2) (I) and (-)-(4) (II) were determined in order to explain the reaction mechanism of the ligand-coupling reaction of (2) to (3) with Grignard reagent.



Experimental. Crystals were obtained from benzene and CH₂Cl₂-hexane solutions for (I) and (II), respectively. Details of data collection and structure refinement are listed in Table 1. Intensity data were collected using a Rigaku AFC-4 diffractometer with graphite monochromator. For both compounds intensities of Bijvoet pairs were measured and treated as independent reflections. Absorption corrections were applied numerically. Structures were solved with *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). H atoms were found from the D maps. Block-diagonal least-squares refinement with anisotropic temperature factors for non-H atoms and isotropic ones for H atoms.

Further refinements were carried out on two enantiomeric structures taking the anomalous-dispersion effect into consideration. $\sum w(|F_o| - k^{-1}|F_c|)^2$ was minimized.

For compound (I), $w = 1/[\sigma^2(F) + 0.01545|F_o| + 0.00022|F_o|^2]$. The final R values for two enantiomers were 0.029 ($wR = 0.040$) and 0.046 ($wR = 0.065$). The former corresponds to the S_S, S_C configuration. For 55 Bijvoet-paired reflections under the conditions of $|F_o| \geq 3.0$, $0.1 \leq (\sin\theta/\lambda) \leq 0.5$ Å⁻¹ and $\Delta F/\langle F \rangle = 2||F(hkl)| - |F(h\bar{k}l)|| / \{|F(hkl)| + |F(h\bar{k}l)|\} \geq 0.1$, R values were 0.033 for S_S, S_C and 0.126 for R_S, R_C . For (II) one of the two independent ClO₄⁻ anions was revealed to be disordered from a D map. Residual peaks around Cl in the D map were assigned as O atoms with the occupancy factors estimated from the peak heights. These atoms were also refined with anisotropic temperature factors. $w = 1/[\sigma^2(F) + 0.01393|F_o| + 0.00205|F_o|^2]$. The final R values were 0.062 ($wR = 0.083$) for the R configuration and 0.076 ($wR = 0.104$) for S . For 315 Bijvoet-paired reflections under the conditions of $|F_o| \geq 5.0$, $0.1 \leq (\sin\theta/\lambda) \leq 0.5$ Å⁻¹ and $\Delta F/\langle F \rangle \geq 0.1$, R values were 0.066 for the R configuration and 0.100 for S .

Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All computations were performed on HITAC M260D and IBM3090-180S computers at the Information Processing Center, University of Electro-Communications, with the programs *UNICSIII* (Sakurai & Kobayashi, 1979) and *MULTAN78* (Main *et al.*, 1978).

Discussion. The final atomic parameters are given in Table 2.* The molecular structures with the atomic numbering are shown in Fig. 1. Bond distances and angles are listed in Table 3.

Structure of (I). The absolute configuration was determined to be S_S, S_C . The geometry of the sulfinyl group has the normal value and is similar to that of (+)-1-phenylethyl 2-quinolyl sulfoxide (Iwasaki, Hirota, Yamazaki & Yasui, 1991). The difference between the lengths of S(1)—C(1) and S(1)—C(2) corresponds to that between S—C_{sp³} and S—C_{sp²}. The selected torsion angles are listed in Table 4 with those of (+)-1-phenylethyl 2-quinolyl sulfoxide. The conformations are also similar to those of the quinolyl derivative except those of the phenyl groups. O(1) is *gauche* to the methyl and phenyl groups

* Lists of structure factors, anisotropic thermal parameters of non-H atoms, atomic parameters for H atoms, and bond lengths and angles for benzene rings of (II) and for ClO₄⁻ anions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54280 (40 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Details of data collection and structure refinement

	(I)	(II)
Colour	Colourless	Colourless
Crystal shape	Needles	Plates
Crystal size (mm)	0.18 × 0.30 × 0.40	0.35 × 0.09 × 0.35
2θ range for cell parameters (°)	65–74	63–75
Number of reflections	25	24
Scan range 2θ (°)	2–130	2–130
Scan width Δω (°)	1.20 + 0.50tanθ	1.15 + 0.45tanθ
Scan speed 2θ (° min ⁻¹)	2	2
Scan mode	2θ-ω	2θ-ω
Monitored reflections (every 50 reflections)	131, 40 $\bar{2}$, 6 $\bar{1}$ 1	32 $\bar{1}$, 105, 4 $\bar{2}$ 3
Variation of intensities	0.976–1.001	0.985–1.001
Range of h, k, l	0–16, –9–9, –6–6	–14–13, –8–8, –18–0
Time for background (s)	10	10
Transmission factor A _{max} –A _{min}	0.725–0.480	0.804–0.441
Number of reflections		
Measured	2277	5272
Unique, R _{int}	2132, 0.014	4952, 0.026
Observed [F _o > 3σ(F)]	2064	4399
R	0.029	0.062
wR	0.040	0.083
Δρ _{max} (e Å ⁻³)	0.199	0.774
(Δ/σ) _{max}	0.040	0.109
S	0.789	1.24

Table 2. Positional parameters (× 10⁵ for S, × 10⁴ for others) and equivalent isotropic temperature factors (Å²) for non-H atoms

	$B_{eq} = (4/3)\sum_i \sum_j \beta_{ij} a_i \cdot a_j$			
	x	y	z	B _{eq}
(I)				
S(1)	34280 (3)	–32287 (10)	37193 (7)	4.37 (1)
O(1)	3014 (1)	–3365 (3)	5999 (2)	6.03 (4)
N(1)	4254 (1)	–730 (2)	1531 (3)	4.88 (4)
C(1)	2437 (1)	–3287 (3)	1403 (3)	4.66 (4)
C(2)	3773 (1)	–1014 (2)	3378 (3)	3.76 (4)
C(3)	3586 (2)	217 (3)	4995 (3)	4.94 (5)
C(4)	3896 (2)	1881 (4)	4632 (4)	5.81 (5)
C(5)	4387 (2)	2218 (3)	2737 (4)	5.43 (6)
C(6)	4550 (2)	893 (3)	1259 (4)	5.53 (6)
C(11)	1727 (1)	–1868 (3)	1636 (3)	4.03 (4)
C(12)	1147 (2)	–1804 (3)	3441 (4)	5.66 (6)
C(13)	504 (2)	–471 (5)	3588 (5)	7.22 (8)
C(14)	411 (2)	781 (4)	1933 (5)	7.12 (8)
C(15)	966 (2)	743 (3)	123 (5)	6.80 (7)
C(16)	1627 (1)	–587 (3)	–47 (4)	5.32 (5)
C(17)	2027 (2)	–5102 (3)	1422 (6)	7.98 (9)
(II)				
C(1A)	–455 (3)	3189 (5)	2726 (2)	3.96 (10)
N(1A)	–1296 (3)	4608 (4)	1265 (2)	3.83 (8)
C(2A)	–1417 (3)	3474 (4)	1880 (2)	3.64 (9)
C(3A)	–2418 (4)	2586 (6)	1679 (3)	4.98 (13)
C(4A)	–3283 (4)	2875 (8)	864 (3)	6.20 (15)
C(5A)	–3122 (4)	4033 (7)	277 (3)	5.97 (14)
C(6A)	–2134 (4)	4894 (6)	485 (3)	5.19 (12)
C(7A)	–224 (4)	5538 (6)	1393 (3)	5.75 (15)
C(11A)	–205 (3)	4791 (4)	3319 (2)	3.72 (9)
C(12A)	–1036 (3)	5904 (5)	3378 (2)	4.22 (10)
C(13A)	–763 (4)	7329 (6)	3957 (3)	4.92 (13)
C(14A)	317 (4)	7620 (6)	4469 (3)	5.71 (14)
C(15A)	1156 (4)	6510 (7)	4422 (3)	6.15 (14)
C(16A)	896 (3)	5091 (7)	3858 (3)	5.12 (12)
C(17A)	–631 (5)	1602 (6)	3240 (3)	5.84 (15)
C(1B)	4787 (3)	3937 (5)	3340 (2)	3.95 (10)
N(1B)	3330 (3)	5425 (5)	2124 (2)	4.96 (10)
C(2B)	4110 (3)	4148 (5)	2363 (2)	3.71 (9)
C(3B)	4265 (3)	3129 (6)	1702 (3)	4.80 (11)
C(4B)	3646 (4)	3351 (8)	811 (3)	6.47 (16)
C(5B)	2862 (4)	4665 (8)	598 (3)	6.87 (16)
C(6B)	2699 (4)	5619 (7)	1227 (4)	6.56 (16)
C(7B)	3162 (5)	6645 (7)	2779 (4)	7.19 (20)
C(11B)	4095 (3)	3140 (5)	3867 (2)	3.78 (9)
C(12B)	3353 (3)	1768 (5)	3539 (3)	4.35 (11)
C(13B)	2776 (4)	1002 (6)	4051 (3)	5.06 (12)
C(14B)	2953 (4)	1584 (7)	4905 (3)	5.47 (14)
C(15B)	3677 (4)	2918 (7)	5242 (3)	5.43 (13)
C(16B)	4243 (3)	3699 (6)	4735 (3)	4.86 (12)
C(17B)	5860 (3)	2898 (7)	3475 (3)	5.51 (13)
O(1A)	704 (1)	188 (1)	1287 (1)	4.64 (3)
O(1A)*	–414 (3)	688 (9)	1003 (3)	10.73 (20)
O(2A)	970 (4)	–509 (6)	2176 (2)	7.87 (14)
O(3A)	841 (4)	–1139 (5)	708 (3)	8.23 (16)
O(4A)	1363 (5)	1643 (6)	1285 (4)	10.40 (21)
O(1B)	5918 (1)	8053 (2)	1885 (1)	5.75 (3)
O(1B)*	5975 (11)	8545 (23)	1148 (7)	17.32 (67)
O(2B)*	6715 (7)	9161 (10)	2585 (5)	10.15 (28)
O(3B)*	6050 (7)	6438 (10)	2256 (10)	14.69 (51)
O(4B)†	4805 (6)	8633 (12)	1958 (7)	10.85 (33)
O(5B)†	7031 (7)	8109 (16)	1637 (7)	7.38 (36)
O(6B)†	5334 (9)	6889 (15)	1040 (6)	6.97 (33)
O(7B)†	5406 (15)	9519 (17)	1595 (14)	12.43 (70)
O(8B)†	6067 (11)	6951 (20)	2582 (7)	9.25 (47)

* Occupancy factor 0.65.

† Occupancy factor 0.35.

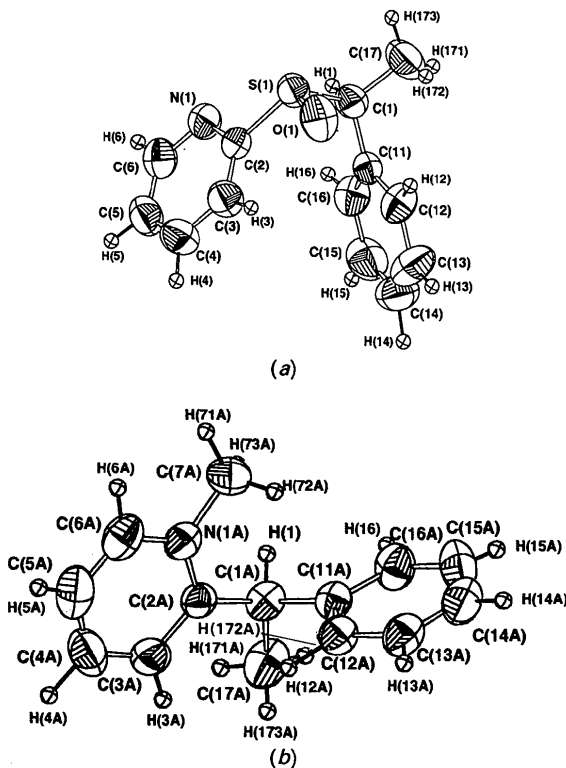


Fig. 1. ORTEP (Johnson, 1976) drawings of (a) (I) and (b) (II), with atom numbering. Thermal ellipsoids for non-H atoms are drawn at 50% probability.

across the S(1)—C(1) bond and *trans* to N(1) across the S(1)—C(2) bond. The torsion angles O(1)—S(1)—C(2)—N(1) of 172.9 (1) and O(1)—S(1)—C(2)—C(3) –3.9 (2)° mean that the S=O bond is nearly in the same plane of the pyridine ring and the

O atom is located far from the non-bonding electron pair of N. The maximum deviation from the pyridyl plane is 0.011 (2) for C(3) and that from the phenyl plane is 0.008 (2) Å for C(12). The dihedral angle between the pyridyl and the phenyl planes is 45.68 (8)°, compared with 38.46 (9)° in the case of quinolyl sulfoxide.

Table 3. Bond lengths (Å) and angles (°) of non-H atoms

(I)			
S(1)—O(1)	1.488 (2)	C(4)—C(5)	1.369 (4)
S(1)—C(1)	1.848 (3)	C(5)—C(6)	1.365 (3)
S(1)—C(2)	1.796 (2)	C(11)—C(12)	1.379 (3)
N(1)—C(2)	1.329 (2)	C(11)—C(16)	1.387 (3)
N(1)—C(6)	1.337 (3)	C(12)—C(13)	1.379 (4)
C(1)—C(11)	1.497 (3)	C(13)—C(14)	1.359 (5)
C(1)—C(17)	1.519 (4)	C(14)—C(15)	1.357 (4)
C(2)—C(3)	1.375 (3)	C(15)—C(16)	1.396 (4)
C(3)—C(4)	1.380 (4)		
O(1)—S(1)—C(1)	108.1 (1)	C(3)—C(4)—C(5)	119.1 (2)
O(1)—S(1)—C(2)	107.1 (1)	C(4)—C(5)—C(6)	118.7 (2)
C(1)—S(1)—C(2)	97.9 (1)	N(1)—C(6)—C(5)	124.2 (2)
C(2)—N(1)—C(6)	115.7 (2)	C(1)—C(11)—C(12)	122.5 (2)
S(1)—C(1)—C(11)	112.6 (2)	C(1)—C(11)—C(16)	119.3 (2)
S(1)—C(1)—C(17)	106.3 (2)	C(12)—C(11)—C(16)	118.2 (2)
C(11)—C(1)—C(17)	114.9 (2)	C(11)—C(12)—C(13)	120.5 (3)
S(1)—C(2)—N(1)	114.0 (1)	C(12)—C(13)—C(14)	120.9 (3)
S(1)—C(2)—C(3)	121.1 (1)	C(13)—C(14)—C(15)	120.0 (3)
N(1)—C(2)—C(3)	124.8 (2)	C(14)—C(15)—C(16)	120.0 (3)
C(2)—C(3)—C(4)	117.5 (2)	C(11)—C(16)—C(15)	120.4 (2)
(II)			
	Mol. A	Mol. B	
C(1)—C(2)	1.504 (5)	1.517 (5)	
C(1)—C(11)	1.524 (5)	1.521 (5)	
C(1)—C(17)	1.527 (7)	1.524 (7)	
N(1)—C(2)	1.357 (5)	1.355 (5)	
N(1)—C(6)	1.358 (6)	1.395 (7)	
N(1)—C(7)	1.483 (6)	1.468 (8)	
C(2)—C(3)	1.377 (6)	1.376 (6)	
C(3)—C(4)	1.412 (8)	1.385 (7)	
C(4)—C(5)	1.353 (8)	1.378 (8)	
C(5)—C(6)	1.353 (7)	1.310 (8)	
C(2)—C(1)—C(11)	112.2 (3)	112.0 (3)	
C(2)—C(1)—C(17)	112.4 (4)	112.2 (3)	
C(11)—C(1)—C(17)	110.9 (4)	110.5 (3)	
C(2)—N(1)—C(6)	121.7 (4)	118.9 (4)	
C(2)—N(1)—C(7)	121.6 (3)	121.7 (4)	
C(6)—N(1)—C(7)	116.6 (4)	119.3 (4)	
C(1)—C(2)—N(1)	119.4 (3)	118.6 (3)	
C(1)—C(2)—C(3)	123.0 (4)	123.2 (4)	
N(1)—C(2)—C(3)	117.5 (4)	118.1 (4)	
C(2)—C(3)—C(4)	120.6 (4)	122.4 (4)	
C(3)—C(4)—C(5)	119.5 (5)	117.5 (5)	
C(4)—C(5)—C(6)	119.2 (5)	120.2 (6)	
N(1)—C(6)—C(5)	121.5 (4)	122.7 (5)	
C(1)—C(11)—C(12)	122.5 (3)	122.3 (3)	
C(1)—C(11)—C(16)	118.7 (4)	120.1 (4)	

Table 4. Selected torsion angles (°)

	(I) S_S, S_C	(R_S, R_C)-Quinoly sulfoxide*
N(1)—C(2)—S(1)—O(1)	172.9 (1)	-170.8 (2)
N(1)—C(2)—S(1)—C(1)	-75.3 (1)	78.2 (2)
O(1)—S(1)—C(1)—C(11)	58.3 (2)	-56.5 (2)
C(2)—S(1)—C(1)—C(11)	-52.7 (1)	53.9 (2)
O(1)—S(1)—C(1)—C(17)	-68.3 (2)	71.3 (2)
C(2)—S(1)—C(1)—C(17)	-179.3 (2)	-178.3 (2)
S(1)—C(1)—C(11)—C(12)	-67.7 (2)	79.7 (2)
S(1)—C(1)—C(11)—C(16)	114.0 (2)	-100.1 (2)
C(17)—C(1)—C(11)—C(12)	54.2 (3)	-43.0 (3)
C(17)—C(1)—C(11)—C(16)	-124.1 (2)	137.3 (2)

* Iwasaki, Hirota, Yamazaki & Yasui (1991).

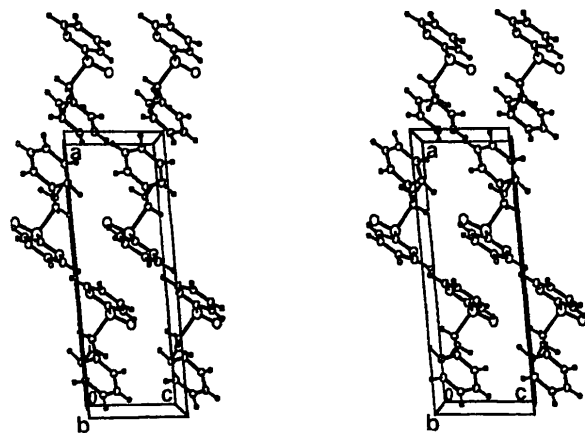
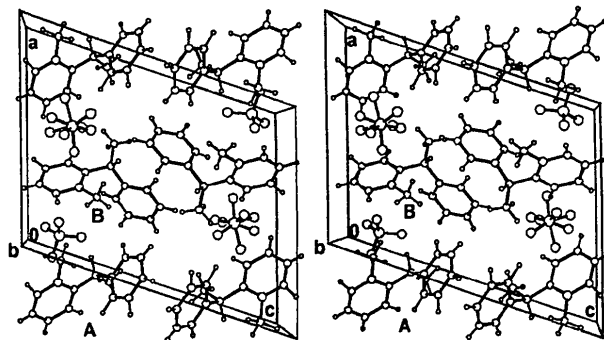
The crystal structure is shown in Fig. 2. The packing mode is that of the van der Waals type.

Structure of (II). The crystal structure is shown in Fig. 3. There are two independent molecules, *A* and *B*, in an asymmetric unit. Consistencies in the bond lengths for both molecules are not so good, however, because of the limit of the analysis due to the disordered perchlorate anion. Slightly shorter

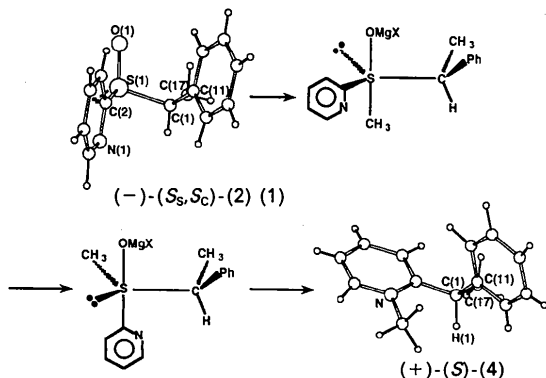
contacts than the sum of van der Waals radii are: 3.049 (8), 3.063 (16) and 2.981 (7) Å for C(2*A*)...O(1*A*), C(2*B*)...O(3*B*) and C(6*A*)...O(3*A*) [(i) $-x, \frac{1}{2} + y, -z$], respectively.

The difference of the conformations about the C(1)—C(2) bond for molecules *A* and *B* is about 7°. The torsion angles across the C(1)—C(11) bond are also different by a similar amount. The pyridinium rings are almost planar with maximum deviations of -0.006 (4) and -0.017 (4) Å for molecules *A* and *B*, respectively. N(1)—C(2) and N(1)—C(6) lengths and C(2)—N(1)—C(6) angles are larger than the corresponding lengths and angles in the pyridine ring of (I). The dihedral angles between the pyridinium and the phenyl planes are 96.6 (2) and 93.5 (2)° for molecules *A* and *B*, respectively.

The absolute configurations are unequivocally determined to be S_S, S_C for (-)-(2) (I) and *R*, for (-)-(4) (II). Since (II) is obtained from the enantiomer of (I), the absolute configuration of the phenylethyl group is retained completely during the reaction from (-)-(2) (I) to (+)-4 shown below. A reaction mechanism, in which the ligand-coupling reaction proceeds between axial and equatorial

Fig. 2. Stereoscopic drawing of (I) viewed along the *b* axis.Fig. 3. Stereoscopic drawing of (II) viewed along the *b* axis.

ligands within the hypervalent σ -sulfurane formed by nucleophilic attack of the Grignard reagent on the sulfinyl S atom, has been proved (Oae *et al.*, 1987).



The authors wish to thank Professor Shigeru Oae, Okayama University of Science, for providing samples and for encouragement. This work was sup-

ported in part by a Grant-in-Aid for Scientific Research on Priority Area (Nos. 01628003 and 02247104) from the Ministry of Education, Science and Culture.

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Absolute Structures of (+)-(R_S,R_C)-1-Phenylethyl 2-Quinolyl Sulfoxide and (-)-(R_C,R_C)-Bis[2-(1-phenylethyl)quinolato]silver Perchlorate

BY FUJIKO IWASAKI,* MASAMICHI HIROTA, NORIMASA YAMAZAKI AND MASONORI YASUI

Department of Applied Physics and Chemistry, The University of Electro-Communications, Chofu-shi, Tokyo 182, Japan

(Received 28 December 1990; accepted 22 May 1991)

Abstract. (I) $C_{17}H_{15}NOS$, $M_r = 281.38$, orthorhombic, $P2_12_12_1$, $a = 10.429$ (2), $b = 16.894$ (2), $c = 8.105$ (2) Å, $V = 1428.2$ (5) Å³, $Z = 4$, $D_x = 1.309$ Mg m⁻³, $\lambda(Cu K\alpha) = 1.54184$ Å, $\mu = 1.912$ mm⁻¹, $F(000) = 592$, $T = 295$ K, $R = 0.046$ for 1992 observed reflections. (II) $C_{34}H_{30}AgN_2^+ \cdot ClO_4^-$, $M_r = 673.94$, orthorhombic, $P2_12_12_1$, $a = 11.686$ (2), $b = 22.938$ (1), $c = 11.364$ (1) Å, $V = 3046.3$ (5) Å³, $Z = 4$, $D_x = 1.470$, $D_m = 1.470$ (5) Mg m⁻³, $\lambda(Cu K\alpha) = 1.54184$ Å, $\mu = 6.550$ mm⁻¹, $F(000) = 1376$, $T = 295$ K, $R = 0.052$ for 4811 observed reflections. The conformations of (I) are similar to those of the corresponding pyridyl compound. In (II) two quinolyl groups are coordinated at Ag with Ag...N distances of 2.195 (4) and 2.192 (5) Å. The N(1A)...Ag...N(1B) angle is 151.6 (2)°. There are weak interactions between the cation and the anion with Ag...O

distances of 2.98 (1) and 2.97 (1) Å. The absolute configurations of these compounds were determined to be R_S, R_C and R_C, R_C for (I) and (II), respectively. The absolute configurations of the phenylethyl groups are retained completely and the reaction mechanism of the ligand coupling *via* hypervalent σ -sulfurane is concluded.

Introduction. When the reaction of (+)-(1) (I) with ethylmagnesium bromide was carried out (-)-2-(1-phenylethyl)quinoline, (-)-(2), was obtained. (-)-(2) was converted to (-)-2-(1-phenylethyl)quinolinium perchlorate, (-)-(3) (Oae, Takeda & Wakabayashi, 1989), in order to introduce a heavy atom to determine the absolute configuration by an X-ray method. The X-ray structure determination of (+)-(1) (I) and (-)-(3) was carried out to explain a reaction mechanism. During the structure analysis, the crystals of the latter compound were revealed not

* To whom all correspondence should be addressed.