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(S-t-BUTYL-N,N'-DIMESITYLSULFURDIIMINE)DICARBONYLRHODIUM, $C_{24}H_{31}N_2O_2RhS$.

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Introduction This investigation was carried out in connection with research into the coordination properties of ambident ligands with a pseudo-allene or pseudo-allyl structure (Kuyper, Keyzer & Vrieze, 1976). The interest was focussed on the coordination of the metal and the conformation of the ligand.

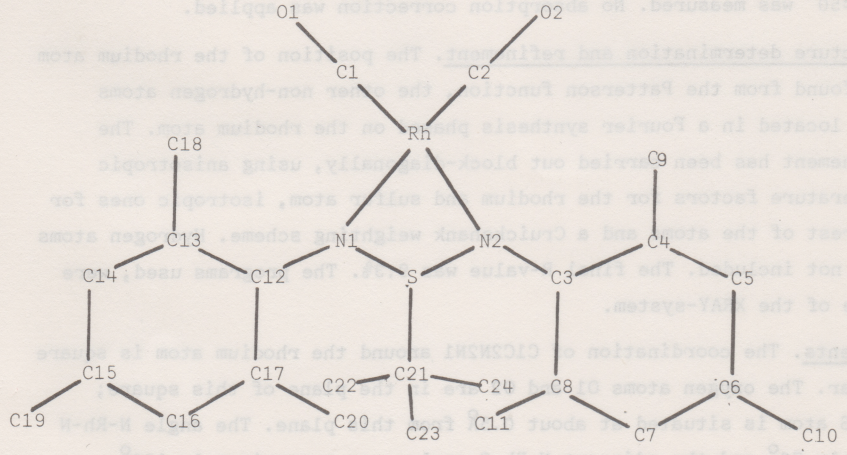
Crystal data. Triclinic with cell constants a=8.439(1), b=8.605(1), c=19.110(3)Å, α=79.41(2), β=83.90(2), γ=63.41(2)°, Z=2, spacegroup $P\bar{1}$. The intensities were collected with a Nonius CAD4 diffractometer using Ni-filtered CuKα-radiation and θ-2θ scan. A total of 2484 reflections up to θ=50° was measured. No absorption correction was applied.

Structure determination and refinement. The position of the rhodium atom was found from the Patterson function, the other non-hydrogen atoms were located in a Fourier synthesis phased on the rhodium atom. The refinement has been carried out block-diagonally, using anisotropic temperature factors for the rhodium and sulfur atom, isotropic ones for the rest of the atoms and a Cruickshank weighting scheme. Hydrogen atoms were not included. The final R-value was 9.3%. The programs used, were those of the XRAY-system.

Comments. The coordination of C1C2N2N1 around the rhodium atom is square planar. The oxygen atoms O1 and O2 are in the plane of this square; the S atom is situated at about 0.5Å from this plane. The angle N-Rh-N is only 70° and the adjacent N-Rh-C angles are approximately 100°. The N-S bond lengths (1.66Å) are longer than was expected, whilst the N-S-N angle is small. With respect to this phenomenon further investigation on analogous compounds is intended.

Fractional coordinates multiplied by 10^4

	x/a	σ	y/b	σ	z/c	σ		x/a	σ	y/b	σ	z/c	σ
Rh	2154	1	4901	1	2122	1	C10	2870	22	-1561	21	5205	9
S	-1230	3	5226	3	2528	1	C11	1961	22	4609	21	4221	9
N1	-0480	11	6303	11	1877	5	C12	-1278	13	7014	13	1191	5
N2	0619	11	4412	11	2965	5	C13	-1431	14	8696	14	0905	6
O1	5719	18	2640	18	2733	7	C14	-2109	16	9429	16	0233	6
O2	3765	17	5980	17	0752	7	C15	-2692	16	8585	15	-0168	6
C1	4332	18	3537	18	2484	7	C16	-2488	15	6934	15	0131	6
C2	3126	18	5556	18	1286	7	C17	-1760	13	6141	13	0793	6
C3	1090	14	2909	14	3511	6	C18	-0913	19	9731	19	1315	8
C4	1070	14	1321	14	3436	6	C19	-3413	18	9439	18	-0892	7
C5	1656	17	-0066	16	3978	7	C20	-1533	18	4210	17	1062	7
C6	2282	17	-0006	17	4613	7	C21	-2910	16	6895	16	3021	6
C7	2343	18	1558	17	4664	7	C22	-2191	21	8122	21	3233	8
C8	1810	16	2973	16	4125	6	C23	-3320	20	5824	20	3709	8
C9	0524	19	1030	19	2747	8	C24	-4500	22	7912	21	2540	9



Bond lengths in Å (standard deviations are approximately .02 for Rh,S - light atom bonds and .03 between light atoms).

Rh	-C(1)	1.81	N(1)	-C(12)	1.44	N(2)	-C(3)	1.43
Rh	-C(2)	1.83	C(12)	-C(13)	1.40	C(3)	-C(4)	1.41
Rh	-N(1)	2.06	C(13)	-C(14)	1.38	C(4)	-C(5)	1.37
Rh	-N(2)	2.06	C(14)	-C(15)	1.40	C(5)	-C(6)	1.39
S	-N(1)	1.66	C(15)	-C(16)	1.37	C(6)	-C(7)	1.39
S	-N(2)	1.65	C(16)	-C(17)	1.38	C(7)	-C(8)	1.38
S	-C(21)	1.83	C(17)	-C(12)	1.36	C(8)	-C(3)	1.40
C(1)	-O(1)	1.17	C(13)	-C(18)	1.51	C(4)	-C(9)	1.54
C(2)	-O(2)	1.18	C(15)	-C(19)	1.49	C(6)	-C(10)	1.51
C(21)	-C(22)	1.56	C(17)	-C(20)	1.58	C(8)	-C(11)	1.51
C(21)	-C(23)	1.56						
C(21)	-C(24)	1.53						

Bond angles in ° (standard deviations are approximately 0.7 for angles involving Rh or S, and 1.3 for those involving light atoms).

C(1)	-Rh	-C(2)	91.1	Rh	-N(1)	-C(12)	129.4	Rh	-N(2)	-C(3)	128.7
C(2)	-Rh	-N(1)	98.9	S	-N(1)	-C(12)	123.2	S	-N(2)	-C(3)	121.9
N(1)	-Rh	-N(2)	70.5	N(1)	-C(12)	-C(13)	115.4	N(2)	-C(3)	-C(8)	116.8
N(2)	-Rh	-C(1)	99.6	N(1)	-C(12)	-C(17)	125.3	N(2)	-C(3)	-C(4)	124.5
Rh	-C(1)	-O(1)	178.3	C(17)	-C(12)	-C(13)	119.2	C(8)	-C(3)	-C(4)	118.3
Rh	-C(2)	-O(2)	179.2	C(12)	-C(13)	-C(14)	118.6	C(3)	-C(4)	-C(5)	119.3
Rh	-N(1)	-S	96.4	C(12)	-C(13)	-C(18)	121.9	C(3)	-C(4)	-C(9)	122.7
Rh	-N(2)	-S	96.6	C(18)	-C(13)	-C(14)	119.5	C(9)	-C(4)	-C(5)	117.8
N(1)	-S	-N(2)	92.1	C(13)	-C(14)	-C(15)	122.9	C(4)	-C(5)	-C(6)	123.5
N(1)	-S	-C(21)	106.0	C(14)	-C(15)	-C(16)	116.2	C(5)	-C(6)	-C(7)	116.0
N(2)	-S	-C(21)	106.5	C(14)	-C(15)	-C(19)	120.6	C(5)	-C(6)	-C(10)	121.5
S	-C(21)	-C(22)	111.5	C(19)	-C(15)	-C(16)	123.2	C(10)	-C(6)	-C(7)	122.5
S	-C(21)	-C(23)	104.6	C(15)	-C(16)	-C(17)	122.3	C(6)	-C(7)	-C(8)	122.5
S	-C(21)	-C(24)	106.4	C(16)	-C(17)	-C(12)	120.8	C(7)	-C(8)	-C(3)	120.1
C(22)	-C(21)	-C(23)	109.4	C(16)	-C(17)	-C(20)	117.1	C(7)	-C(8)	-C(11)	118.9
C(23)	-C(21)	-C(24)	112.7	C(20)	-C(17)	-C(12)	122.1	C(11)	-C(8)	-C(3)	121.0
C(22)	-C(21)	-C(24)	112.0								

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Reference.

Kuyper, J. Keijzer, P.C. and Vrieze, K.; (1976) J. Organometal. Chem., to be published.

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