

Research Article

Neutral adducts of Arylorganoantimony (III) Chlorides, Ar_nSbCl_{3-n} with Oxygen, Nitrogen and Sulphur Donors

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ABSTRACT

Tetra & Penta-coordinate neutral adducts $(C_6H_5)_2SbCl.L$ and $(C_6H_5)SbCl_2.2L$ [$L = \alpha, \beta$ & γ -picoline, HMPA, Ph_3PO , TU, Py] have been synthesized. Molecular adducts are monomeric in benzene and non-electrolyte in acetonitrile. IR spectra and conductance measurement suggest the presence of coordination of Oxygen, Nitrogen, and Sulphur donor ligands in adducts. Spectroscopic data conform to the requirement of tetra coordinate and penta coordinate configuration for neutral adducts.

Keywords: Diaryl & arylantimony (III)chloride; molecular adducts; monomeric; non-electrolyte.

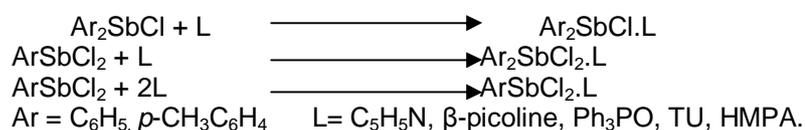
INTRODUCTION

Lewis acidity of antimony (III) halides towards electronegatively charged donors to give $[SbX_4]^{-4}$, $[SbX_5]^{-2}$, $[SbX_6]^{-3}$; towards neutral donors such as phosphine oxides and arsine oxides and crown ether is well known¹⁻⁷. However, complex formation between organoantimony (III) halides and neutral donors is less well established. Nunn et al.⁷ recently reported some representative complexes of $SbPhX_2$ and $SbPh_2X$ (where $X = Cl, Br, I$) with nitrogen donors, viz., pyridine, 2, 2'-bipyridyl and 1, 10 phenanthroline. It was observed that with monophenylantimony(III) halides, pyridine gives both 1:1 and 1:2 complexes while with diphenylantimony(III) halides only 1:1 complexes are formed. Bidentate ligands form 1:1 complexes both with monophenyl and diphenyl-antimony (III) halides. A perusal of literature reveals that neutral complexes of $SbPh_nX_{3-n}$ with oxygen or sulphur donors have not been reported to

date^{6,7}. A series of hitherto unreported molecular adducts of Ar_nSbCl_{3-n} ($n=1,2$) with O, N and sulphur donors (Py, β -pic, Ph_3PO , HMPA and TU) in 1:1 or 1:2 stoichiometry [$Ar_2SbCl.L$; $ArSbCl_2.L$ and $ArSbCl_2.2L$] has been synthesized and characterized.

RESULTS AND DISCUSSION

As expected, arylantimony (III) halides readily give Lewis acid-base addition compounds. With monoarylantimony (III) chloride, pyridine, β -picoline, TU, HMPA and triphenylphosphineoxide from both 1:1 and 1:2 complexes, but with the corresponding diarylantimony(III) chloride only 1:1 complexes are formed, perhaps because of the lower Lewis acidity of Ar_2SbCl derivatives. The complexes are conveniently prepared by the direct interaction of the reactants in any anhydrous chloroform solution.



The complexes are white or yellow crystalline solids. Complexes with Ar_2SbCl are somewhat difficult to crystallize on account of lesser Lewis acidity. The complexes were crystallized from chloroform or dichloromethane. They are hydrolytically stable and can be stored at room temperature without decomposition.

The molar conductance values of 10^{-3} M solutions in acetonitrile show that these complexes behave as non-electrolytes in solution. Molecular weight data in

nitrobenzene indicate them to be monomeric, the elemental analysis and other data are collected in Tables-1, 2, 3 & 4 and are consistent with the proposed formulation of the complexes.

Infrared spectra

All the complexes (listed in table 1) were characterized in the solid state by their infrared spectra in the region $4000\text{-}200\text{cm}^{-1}$. Important IR frequencies for the complexes

together with their assignment are listed in table-4. Their assignment has been made by comparing the spectra in $4000-200\text{cm}^{-1}$ region in the solid state of the complexes with those of free ligands.

The IR absorption due to C_6H_5 group attached to antimony atom are almost identical and do not differ significantly from those observed for pentafluorophenyl (V) compounds earlier^{2,3,4}. Infrared absorption for all the isolated compounds associated with the antimony aryl groups and the aromatic donors overlap and present a complex picture which is considerably difficult to make proper assignment. However, bands due to aromatic donors which are characteristic have been identified to a certain extent. Thus, pyridine out of plane deformation⁶ observed at 404cm^{-1} in the free ligand, is shifted to 415 ± 5 in (p-tolyl) SbCl_2 and (p-tolyl)₂ SbCl complexes. Similarly, in plane deformation mode which appears at 604cm^{-1} in the free ligand is shifted to $620\pm 2\text{cm}^{-1}$ in the 1:1 and 1:2 complexes. The characteristic $\nu\text{C}=\text{N}$ vibration in the adducts with nitrogen donors (pyridine and β -picoline) reported to appear in the range $1568-1575\text{cm}^{-1}$ in free ligands¹¹ undergoes a positive shift on complexation and appears at $1600\pm 15\text{cm}^{-1}$ suggesting coordination through nitrogen atom of the base.

Similarly, in the case of oxygen donor, Ph_3PO , $\nu(\text{P}-\text{O})$ appearing at 1192cm^{-1} in the free ligand show a negative shift and appear around 1125 ± 5 indicating coordination through oxygen atom of the ligand¹¹.

In case of HMPA complexes, the characteristic $\nu(\text{P}=\text{O})$ vibrations appearing at 1212cm^{-1} in free ligand is considerably lowered to $1138\pm 2\text{cm}^{-1}$ on complexation indicating coordination through oxygen atom of the base¹⁰.

In case of thiourea complexes, $\nu\text{C}=\text{N}$ and $\nu\text{C}=\text{S}$ do not suffer any significant shift compared to free ligand. The positive shift of $\nu(\text{N}-\text{H})$ from 3300cm^{-1} in free thiourea to 3370cm^{-1} in the adducts indicates coordination through nitrogen atom of the ligand¹¹.

As expected, bands associated with antimony chlorine modes move to lower energy on complexation, from 340 to 320cm^{-1} in SbArCl_2 to $310\pm 5\text{cm}^{-1}$ in the complexes. In case of Ar_2SbCl band appearing at 305cm^{-1} is shifted to $280\pm 10\text{cm}^{-1}$, $\text{Sb}-\text{C}$ bond appears in the range $450-470\text{cm}^{-1,2,6}$.

Thus on the basis of IR spectra, molecular weight and conductance measurements, the neutral molecular adducts can be assigned tetra coordinate and penta coordinate structure as shown below. Similar structures have earlier been assigned for 1:1 and 1:2 addition products with Ph_2SbCl and PhSbCl_2 , respectively^{2,6,11 & 12}.

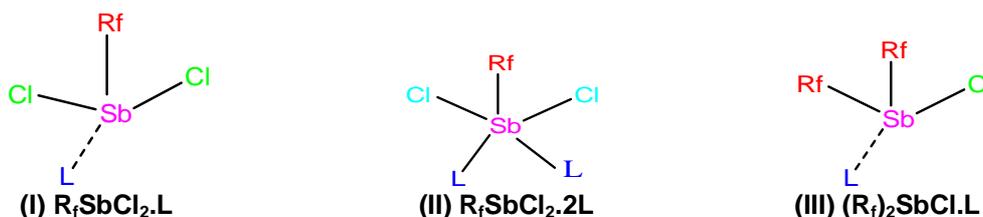
Stereochemistry of neutral molecular adducts $(\text{Ar})_2\text{SbCl}$ L & $\text{ArSbCl}_2.2\text{L}$

It has been assumed that the addition of Lewis base, L to the central atom in a tetra coordinate & penta coordinate molecule takes place in a steric and electrostatic factor play an important role in determining the position of entry of L. It is well established that the more electronegative group goes to the axial position and less electronegative on equatorial positions. Therefore base L should settle in the equatorial position.

In view of the above idea the nucleophilic attack at position between two chloro groups to produce structure appears to be most favourable, since Rf is less electronegative than any halogen atom directly bonded to metal.

Thus analytical, conductance measurement, molecular weight determination and IR data clearly indicates that the newly synthesized complexes have tetra & penta co-ordination environment around antimony with tetrahedral configuration as has been suggested for $\text{Ar}_2\text{SbCl.L}$ complexes^{5,12}. It is generally accepted that the diarylantimony (III)chloride have a geometry of a tetrahedral.

In adduct formation as indicated, antimony atom increases its coordination number to four & fifth for penta coordinate complexes. A tentative assignment of tetrahedral structure may be represented as below (Fig. 1).



$\text{R}_f = (p\text{-CH}_3\text{C}_6\text{H}_4)$, $\text{C}_6\text{H}_5\text{Sb}$; $\text{L} = \text{Pyridine}(\text{C}_5\text{H}_5\text{N})$, α, β, γ -picoline, triphenylphosphine oxide(Ph_3PO), HMPA, Thiourea(H_2NCSNH_2)

¹H NMR spectra

The ¹H NMR spectra of compound no I exhibit one singlet at δ 0.90 ppm most downfield (aldehydic proton), a triplet for methyl proton at δ 2.96 ppm most shielded, a sextet for methylene protons at δ 1.60 ppm and a triplet at δ 2.96 ppm for methylene group attached with nitrogen atom are characteristics for DPF complex. Compound No IV, exhibits most shielded peak for protons of methyl group ranges from δ 7.18 ppm to δ 8.69 ppm are characteristics for picoline complexes. Compound no X exhibits multiple range δ 7.3 ppm to δ 7.77 ppm for proton of phenyl group attached to phosphorous atom are characteristics for triphenylphosphine oxide complexes. Compound No XXIV exhibit a singlet at δ 9.53 ppm for protons attached at amine groups are characteristics of thiourea complexes. All the ¹H NMR spectra of aforesaid compounds are taken in CDCl₃ as solvent and tetramethylsilane as a reference material.

Experimental

Preparation of (p-CH₃C₆H₄)₂SbCl₂.Py (I)

To a stirring solution of di (p-tolyl) antimony (III) chloride (0.339 g; 1 mmol) in dry chloroform (30 ml), a solution of pyridine (0.079 g; 1 mmol) in ethanol (30 ml) was added drop wise. The reactants were stirred for 4 h at room temperature and then refluxed for one hour to ensure complete reaction. It was then filtered, and the filtrate was distilled off to remove excess of solvent to get cream colour solid of di(p-tolyl) antimony chloride pyridine. It was recrystallized from petroleum-ether (40 – 60°C).

Preparation of (C₆H₅)₂SbCl₂. β -Picoline (IV)

A solution of (C₆H₅)₂SbCl (0.311 g, 1 mmol) in chloroform (30 ml) and β -Picoline (0.093 g, 1 mmol) were stirred together at room temperature for 5 h in nitrogen atmosphere. It

was filtered off and the filtrate on concentration in vacuo afforded a white solid of diphenyl antimony(III) chloride β -picoline adduct, which was recrystallized by petroleum-ether (40 - 60°C).

Preparation of C₆H₅SbCl₂.2Ph₃PO (X)

To a stirring solution of phenylantimony(III) dichloride (0.270 g; 1 mmol) in chloroform (30 ml) was added dropwise triphenylphosphine oxide (0.556 g; 2 mmol). The reactants were stirred for 4 h at room temperature under nitrogen and then refluxed further for 2 hour. It was then filtered, and the filtrate on concentration in vacuo yielded a white solid characterized as phenylantimony(III) dichloride triphenylphosphine oxide. It was recrystallized from benzene.

Preparation of p-CH₃C₆H₄SbCl₂.HMPA (XVI)

To a stirring solution of p-CH₃C₆H₄SbCl₂ (0.284 g; 1 mmol) in chloroform (30 ml) was added HMPA (0.179 g; 1 mmol). In ethanol were stirred together at room temperature for 6 h under nitrogen. It was then filtered, and the filtrate on crystallised in vacuo afforded a white solid which was recrystallised from petroleum ether (40-60°C) to give the (p-tolyl)antimony(III) dichloride HMPA.

Preparation of C₆H₅SbCl₂.Thiourea (XXII)

To a stirring solution of diphenylantimony(III) chloride (0.311 g; 1 mmol) in chloroform (30 ml) thiourea(0.076 g; 1 mmol) in methanol(30ml) was added drop wise.. The reactants were stirred for 6 h at room temperature under nitrogen and then refluxed further for 1 hour to ensure complexation of reaction. It was filtered off, and the filtrate on concentration in *vacuum* yielded a white solid. It was recrystallized from petroleum ether (40-60°C) to get diphenylantimony(III)chloride thiourea.

Table 1: Preparation, properties and elemental analysis of Neutral Molecular Adducts of diaryl and aryl antimony(III) chloride

| C.No | Adducts/Complexes | Ligand (g) Solvent (ml) | M.P. (°C) | Colours | Found (Calcd)% | | |
|-------|--|--|------------------|---------|----------------|------------|--------------|
| | | | | | C | H | N |
| | | L=Pyridine | | | | | |
| I | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl.C ₅ H ₅ N | C ₅ H ₅ N(0.079g).EtOH | 100 | Cream | 54.28(54.48) | 4.44(4.54) | 3.21(3.34) |
| II | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl ₂ .C ₅ H ₅ N | C ₅ H ₅ N(0.079g).EtOH | 130 | Cream | 39.59(39.66) | 3.24(3.30) | 3.79(3.85) |
| III | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl ₂ .2C ₅ H ₅ N | C ₅ H ₅ N(0.158g).EtOH | 150 | Cream | 46.09(46.15) | 3.76(3.85) | 6.24(6.33) |
| | | L=picoline | | | | | |
| IV | (C ₆ H ₅) ₂ SbCl.C ₆ H ₇ N | β-C ₆ H ₇ N(0.093g)EtOH | 170 | White | 52.70(53.40) | 4.11(4.20) | 3.40(3.46) |
| V | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl.C ₅ H ₇ N | β-C ₆ H ₇ N (0.093g)EtOH | 115 | White | 54.60(55.50) | 4.78(4.85) | 3.18(3.23) |
| VI | C ₆ H ₅ SbCl ₂ .C ₆ H ₇ N | β-C ₆ H ₇ N (0.093g)EtOH | 120 | Cream | 39.59(39.66) | 3.21(3.30) | 3.78(3.85) |
| VII | C ₆ H ₅ SbCl ₂ .2C ₆ H ₇ N | β-C ₆ H ₇ N (0.186g)EtOH | 140 | Cream | 47.29(47.36) | 4.10(4.16) | 6.08(6.14) |
| VIII | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl ₂ .C ₅ H ₇ N | β-C ₆ H ₇ N (0.093g)EtOH | 125 | Cream | 41.30(41.37) | 3.64(3.71) | 3.54(3.17) |
| IX | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl ₂ .2C ₅ H ₇ N | β-C ₆ H ₇ N (0.186g)EtOH | 150 | Cream | 47.95(48.50) | 4.40(4.46) | 5.87(5.95) |
| | | L=Ph₃PO | | | | | |
| X | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl.Ph ₃ PO | (C ₆ H ₅) ₃ PO(0.278g)EtOH | 100 | White | 62.10(62.18) | 4.61(4.69) | - |
| XI | (C ₆ H ₅) ₂ SbCl.Ph ₃ PO | (C ₆ H ₅) ₃ PO(0.278g)EtOH | 110 | White | 60.90(61.00) | 4.17(4.24) | - |
| XII | C ₆ H ₅ SbCl ₂ .Ph ₃ PO | (C ₆ H ₅) ₃ PO(0.278g)EtOH | 170 | White | 52.00(52.50) | 2.90(3.60) | - |
| XIII | C ₆ H ₅ SbCl ₂ .2Ph ₃ PO | (C ₆ H ₅) ₃ PO(0.556g)EtOH | 115 | White | 60.80(61.00) | 4.10(4.23) | - |
| XIV | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl ₂ .Ph ₃ PO | (C ₆ H ₅) ₃ PO(0.278g)EtOH | 95 | White | 52.70(53.30) | 3.00(3.90) | - |
| XV | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl ₂ .2Ph ₃ PO | (C ₆ H ₅) ₃ PO(0.556g)EtOH | 150 | Off | 60.00(61.40) | 4.25(4.40) | - |
| | | L=HMPA | | | | | |
| XVI | <i>p</i> -CH ₃ C ₆ H ₄ SbCl ₂ .HMPA | [(CH ₂ N] ₃ P(O))(0.179g)EtOH | 200 | White | 32.00(33.70) | 4.00(5.40) | 8.80(9.07) |
| XVII | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl ₂ .2HMPA | [(CH ₂ N] ₃ P(O))(0.358g)EtOH | 215 ^d | Off | 34.20(35.50) | 5.50(6.70) | 11.90(13.00) |
| XVIII | C ₆ H ₅ SbCl ₂ .HMPA | [(CH ₂ N] ₃ P(O))(0.179g)EtOH | 180 | White | 31.10(32.10) | 4.95(5.12) | 9.15(9.35) |
| XIX | C ₆ H ₅ SbCl ₂ .2HMPA | [(CH ₂ N] ₃ P(O))(0.358g)EtOH | 100 | White | 33.3(34.40) | 6.40(6.53) | 12.8(13.40) |
| XX | (C ₆ H ₅) ₂ SbCl.HMPA | [(CH ₂ N] ₃ P(O))(0.179g)EtOH | 175 | Off | 42.10(44.00) | 5.65(5.71) | 8.10(8.56) |
| XXI | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl.HMPA | [(CH ₂ N] ₃ P(O))(0.179g)EtOH | 120 | White | 45.80(46.30) | 5.95(6.17) | 7.65(8.10) |
| | | L=Thiourea.H₂NCSNH₂ | | | | | |
| XXII | (C ₆ H ₅) ₂ SbCl.H ₂ NCSNH ₂ | H ₂ NCSNH ₂ (0.076g)MeOH | 230 ^d | White | 39.80(40.20) | 3.40(3.61) | 7.01(7.22) |
| XXIII | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl.H ₂ NCSNH ₂ | H ₂ NCSNH ₂ (0.076g).MeOH | 136 | Yellow | 42.80(43.30) | 3.90(4.30) | 5.85(6.70) |
| XXIV | C ₆ H ₅ SbCl ₂ .H ₂ NCSNH ₂ | H ₂ NCSNH ₂ (0.076g).MeOH | 110 | Yellow | 24.10(24.27) | 2.50(2.60) | 7.90(8.09) |
| XXV | C ₆ H ₅ SbCl ₂ .2H ₂ NCSNH ₂ | H ₂ NCSNH ₂ (0.152g).MeOH | 150 | Yellow | 21.60(22.70) | 2.90(3.08) | 12.80(13.20) |

Table 2: Molecular weight, Conductance measurement & Yield of Neutral Molecular adducts of diaryl and arylantimony (III) chloride

| C.No | Adducts /Complexes | Molar conductance (Ohm ⁻¹ cm ² mol ⁻¹) acetonitrile | Molecular weight in nitrobenzene Found (calcd). | Yield | |
|-------|--|---|---|-------|----|
| | | | | g | % |
| I | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl.C ₅ H ₅ N | 2.4 | 416(418.5) | 0.29 | 71 |
| II | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl ₂ .C ₅ H ₅ N | 5.8 | 350(363) | 0.27 | 74 |
| III | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl ₂ .2C ₅ H ₅ N | 3.2 | 434(442) | 0.32 | 72 |
| IV | (C ₆ H ₅) ₂ SbCl.C ₆ H ₇ N | 4.7 | 397(404) | 0.30 | 75 |
| V | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl.C ₅ H ₇ N | 2.3 | 430(432.5) | 0.31 | 73 |
| VI | C ₆ H ₅ SbCl ₂ .C ₆ H ₇ N | 6.2 | 360(363) | 0.28 | 76 |
| VII | C ₆ H ₅ SbCl ₂ .2C ₆ H ₇ N | 8.9 | 455(456) | 0.34 | 75 |
| VIII | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl ₂ .C ₅ H ₇ N | 3.5 | 367(377) | 0.29 | 76 |
| IX | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl ₂ .2C ₅ H ₇ N | 7.2 | 467(470) | 0.33 | 70 |
| X | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl.Ph ₃ PO | 5.8 | 615.5(617.5) | 0.47 | 76 |
| XI | (C ₆ H ₅) ₂ SbCl.Ph ₃ PO | 6.4 | 586.5(589.5) | 0.44 | 74 |
| XII | C ₆ H ₅ SbCl ₂ .Ph ₃ PO | 5.9 | 547(548) | 0.38 | 70 |
| XIII | C ₆ H ₅ SbCl ₂ .2Ph ₃ PO | 2.3 | 824(824) | 0.57 | 69 |
| XIV | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl ₂ .Ph ₃ PO | 5.7 | 559(562) | 0.40 | 72 |
| XV | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl ₂ .2Ph ₃ PO | 3.1 | 838(840) | 0.59 | 71 |
| XVI | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl ₂ .HMPA | 4.6 | 461(462) | 0.33 | 72 |
| XVII | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl ₂ .2HMPA | 2.2 | 638(642) | 0.47 | 73 |
| XVIII | C ₆ H ₅ SbCl ₂ .HMPA | 6.1 | 447(449) | 0.29 | 66 |
| XIX | C ₆ H ₅ SbCl ₂ .2HMPA | 8.8 | 625(628) | 0.42 | 67 |
| XX | (C ₆ H ₅) ₂ SbCl.HMPA | 3.6 | 488.5(490.5) | 0.32 | 66 |
| XXI | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl.HMPA | 7.1 | 515.5(518.5) | 0.35 | 67 |
| XXII | (C ₆ H ₅) ₂ SbCl.H ₂ NCSNH ₂ | 5.7 | 385.5(387.5) | 0.23 | 59 |
| XXIII | (<i>p</i> -CH ₃ C ₆ H ₄) ₂ SbCl.H ₂ NCSNH ₂ | 6.3 | 413.5(415.5) | 0.26 | 62 |
| XXIV | C ₆ H ₅ SbCl ₂ .H ₂ NCSNH ₂ | 5.7 | 342(346) | 0.22 | 64 |
| XXV | C ₆ H ₅ SbCl ₂ .2H ₂ NCSNH ₂ | 6.7 | 423(422) | 0.26 | 63 |

Table 3: InfraRed spectra for Neutral Molecular adducts of diaryl and arylantimony (III) chloride
 $(Ar)_nSbCl_{3-n}$ ml cm^{-1} [n=1;m=2;n=2;m=1]

| Compd. No. (Adduct) | v(Sb-Cl) | v(Sb-I) / v(Sb-N) | v(C=N)/v(P-O) / v(N-H) / v(As-O) ligand (complex) |
|---------------------|----------|-------------------|---|
| I | 290ms | 370w | 404,604,1568 |
| II | 310 | 381w | 404,604,1568 (419,618,1585) |
| V | 289s | 386w | 419,621,1610 |
| IX | 292s | 390w | 880(840) |
| XI | 288s | 419w | 1192(1130) |
| XII | 316s | 415w | 1120 |
| XIII | 292s | 424w | 1212(1140) |
| XV | 315s | 432w | 1212(1136) |
| XVI | 290s | - | 3300(3368) |
| XVIII | 310s | - | 3300(3380) |

w = weak, ms = medium strong, s = strong

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