

III. Infrared Spectroscopy. Infrared spectra were obtained (Table VI) in order to further assess the value of the CP/MAS technique for solid-state structure determination. The observation of single ν_{CN} vibrations is consistent with the conclusion based upon NMR data that all the cyanide complexes are trans in the solid state. The infrared spectra of all the azide complexes showed two bands corresponding to the symmetric and asymmetric ν_{N_3} stretching modes expected for cis isomers. However, the infrared data for the halide complexes are not as informative. Three of the iodides show single ν_{MI} vibrations in the observed region

whereas the NMR data show the presence of both the cis and trans isomer in the solid state for two of these complexes. Similarly, for all the chlorides, only two ν_{MCl} vibrations could be identified, and for the palladium complexes, three are expected. The expected number of ν_{MBr} vibrations is observed in each case. As a consequence, the infrared data are sometimes ambiguous.

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A New Yttrium Sesquichloride Nitride, $\beta\text{-Y}_2\text{Cl}_3\text{N}$, That Is Isostructural with the Binary Yttrium Sesquichloride

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Reactions of YCl_3 , YN , and Y metal in sealed Nb containers over a range of temperatures and compositions afford $\beta\text{-Y}_2\text{Cl}_3\text{N}$, whereas only $\alpha\text{-Y}_2\text{Cl}_3\text{N}$ in the $(\alpha\text{-})\text{Gd}_2\text{Cl}_3\text{N}$ structure is obtained in the absence of metal. $\beta\text{-Gd}_2\text{Cl}_3\text{N}$ is synthesized similarly. The $\beta\text{-Y}_2\text{Cl}_3\text{N}$ ($\beta\text{-Gd}_2\text{Cl}_3\text{N}$) gives a powder pattern that is indistinguishable from that of the infinite cluster chain phase Y_2Cl_3 (Gd_2Cl_3) except for small increases in lattice parameters corresponding to a 2% (1%) increase in cell volume. ($\alpha\text{-Y}_2\text{Cl}_3\text{N}$: $a = 12.761$ (1) Å, $b = 6.676$ (2) Å, $c = 6.1000$ (7) Å (*Pbcn*). $\beta\text{-Y}_2\text{Cl}_3\text{N}$: $a = 15.248$ (2) Å, $b = 3.8520$ (4) Å, $c = 10.157$ (2) Å, $\beta = 118.41^\circ$ (*C2/m*, $Z = 4$). $\beta\text{-Gd}_2\text{Cl}_3\text{N}$: $a = 15.290$ (5) Å, $b = 3.912$ (1) Å, $c = 10.209$ (3) Å, $\beta = 117.79$ (3)°.) A single-crystal X-ray study of $\beta\text{-Y}_2\text{Cl}_3\text{N}$ ($R = 3.9\%$, $R_w = 4.8\%$) demonstrates that the nitrogen atoms are positioned in tetrahedral metal sites above and below the shared metal edges of the infinite chain of octahedra found in Y_2Cl_3 . The need for metal in the syntheses, the black color of the fibrous crystals, and the refined occupancy of nitrogen (81 (2)%) in the yttrium phase all suggest a small nitrogen deficiency in $\beta\text{-(Y,Gd)}_2\text{Cl}_3\text{N}$. The close correspondence of distances in the binary vs the ternary (β) sesquichlorides, particularly the small (0.024 Å) increase in the shared Y–Y edge, is discussed.

Introduction

The first window on a new chemistry of metal-rich halides was the discovery of gadolinium sesquichloride² and the demonstration that its remarkable structure contained infinite chains constructed of metal octahedra, as could be generated by condensing Gd_6Cl_8 -type clusters through sharing of trans edges.³ Since then, several isostructural sesquihalides have been described (Y_2Cl_3 , Gd_2Br_3 , Tb_2Cl_3),^{4,5} and Gd_2Cl_3 has been shown to be a semiconductor.⁶ All of these phases appear to be true binary compounds free of essential interstitial atoms.

More recently, a much larger array of ternary halides has been discovered that require an interstitial atom within each metal octahedron for stability. The employment of carbon with the gadolinium halides provides a variety of such compounds⁷ but with compositions and structure types other than that of Gd_2Cl_3 . For reduced yttrium chloride systems, only the layered $\text{Y}_2\text{Cl}_2\text{C}$ ⁸ and $\text{Y}_3\text{Cl}_3\text{C}$ ⁹ are known to contain carbon atoms within condensed Y_6 octahedra.

In contrast, the utilization of a nitrogen source in syntheses generally does not yield compounds comparable to those observed with carbon. Related to the above sesquihalide structure are the cluster chains found in $\text{Sc}_4\text{Cl}_6\text{N}$ ¹⁰ and $\text{Sc}_3\text{Cl}_8\text{N}$ ¹¹ in which ni-

trogen-centered $\text{Sc}_6\text{Cl}_{12}$ -type clusters also share trans metal edges. On the other hand, scandium products are usually different from those formed by any other rare-earth element, and analogues are not known elsewhere. Another nitride structure type is found in $\text{Gd}_2\text{Cl}_3\text{N}$ ¹² in which nitrogen-containing gadolinium *tetrahedra* are linked via shared opposite edges to again form infinite chains. But the result is now a gadolinium(III) compound with empty gadolinium d bands¹³ and therefore negligible metal–metal bonding. More recently, the nitrogen-poorer analogue $\text{Gd}_3\text{Cl}_6\text{N}$ has been synthesized and shown to contain isolated pairs of Gd_4N tetrahedra that share a common edge.¹⁴

Our exploration of the role of nitrogen in reduced yttrium chlorides has yielded not only the known Y_2Cl_3 and $\alpha\text{-Y}_2\text{Cl}_3\text{N}$ isostructural with the above $(\alpha\text{-})\text{Gd}_2\text{Cl}_3\text{N}$ but also a good yield of a new phase with a powder pattern in which both the line positions and their intensities correspond very well to those of a slightly expanded Y_2Cl_3 structure. X-ray studies reported here show that this $\beta\text{-Y}_2\text{Cl}_3\text{N}$ is isostructural with the binary phase Y_2Cl_3 save for introduction of nitrogen atoms in tetrahedral metal sites, not in the cluster-centered octahedral positions. Questions regarding the presence of metal–metal bonding and the compound's valence character, already discussed for $(\alpha\text{-})\text{Gd}_2\text{Cl}_3\text{N}$, arise again for the new $\beta\text{-Y}_2\text{Cl}_3\text{N}$.

Experimental Section

Synthesis. High-purity, sublimed yttrium metal (Johnson Matthey, 99.997%) was cut into small pieces ($\sim 3 \times 3$ mm) prior to use. Yttrium trichloride was prepared from the reaction of Y_2O_3 (Ames Laboratory) with NH_4Cl .¹⁵ The resulting $(\text{NH}_4)_3\text{YCl}_6$ was decomposed to YCl_3 at 400 °C under vacuum, and the product was sublimed twice at 870 °C and $\sim 10^{-5}$ Torr in a tantalum tube within a silica jacket. YN was synthesized¹⁶ by first hydriding the metal in a molybdenum boat at 560

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Table I. Crystal Data for β -Y₂Cl₃N

space group; Z	C2/m (No. 12); 4	μ (Mo K α), cm ⁻¹	254.1
a, Å ^a	15.238 (2)	transm factor:	0.996; 0.285
b, Å	3.8535 (4)	max; min	
c, Å	10.156 (2)	R ^b	0.039
β , deg	118.38 (2)	R _w ^c	0.048
V, Å ³	524.7 (5)		

^a Diffractometer data. ^b $R = \sum ||F_o| - |F_c|| / \sum |F_o|$. ^c $R_w = [\sum w(|F_o| - |F_c|)^2 / \sum w|F_o|^2]^{1/2}$; $w = 1/\sigma(|F_o|^2)$.

°C under slightly less than 1 atm of H₂. After hydrogen uptake, the temperature was raised to 900 °C, and slightly less than 1 atm of dry N₂ was introduced for 5 h. The X-ray powder data from the product showed the broad pattern of cubic YN and no extra lines.

Reactions of YCl₃, YN, and Y metal were performed in welded niobium ampules as described before.⁸ The phases are characteristically very moisture sensitive and therefore were handled only in a glovebox. Starting materials as well as products were identified by X-ray powder means.

α -Y₂Cl₃N. The stoichiometric 1:1 mixture of YCl₃ and YN was reacted at 850 °C for 10 days. The air-quenched product contained approximately 70% of the brown, powdered α -Y₂Cl₃N (Gd₂Cl₃N structure¹²), some unreacted YCl₃ and YN, and a trace of YOCl. Further experiments showed that α -Y₂Cl₃N could be obtained in a temperature range between 750 and 1000 °C. An isomorph of Gd₃Cl₆N¹⁴ was not seen.

β -Y₂Cl₃N. Black fibrous needles of β -Y₂Cl₃N were obtained by allowing YCl₃, YN, and Y metal to react. Typical reactions were loaded with 130 mg of YCl₃, 35 mg of YN, and 30 mg of Y powder, corresponding to the stoichiometry Y₄Cl₆N, and were heated to 900 °C for 2 days followed by 800 °C for 14 days. The quenched product contained 60–70% β -Y₂Cl₃N besides unreacted YCl₃, a little α -Y₂Cl₃N, and a trace of YOCl. Representative amounts of β -Y₂Cl₃N were obtained at temperatures between 800 and 900 °C. However, the new phase was never seen after reactions of stoichiometric proportions of YCl₃ and YN alone. Reactions with overall stoichiometries between Y₄Cl₆N and Y₆Cl₉N were most suitable, indicating the requirement of a certain excess of metal or a deficiency of nitrogen. Analogues of known scandium phases such as Y₂Cl₂N, Y₄Cl₆N, Y₃Cl₅N, or Y₇Cl₁₂N were not seen even from reactions loaded for these compositions. The comparable β -Gd₂Cl₃N can be readily synthesized under similar conditions.

The products were characterized by X-ray powder pattern (monochromated Cu K α radiation, $\lambda = 1.54056$ Å) secured with an Enraf-Nonius (FR-552) Guinier camera and NBS silicon powder as an internal standard. Lattice parameters for α -Y₂Cl₃N ($a = 12.761$ (1) Å, $b = 6.676$ (2) Å, $c = 6.1000$ (7) Å, from 31 to 60 indexed lines) were calculated on the basis of the indexing for the isostructural Gd₂Cl₃N (*Pbcn*). Dimensional calculations for β -Y₂Cl₃N based on up to 62 lines indexed according to the Y₂Cl₃ structure gave slightly expanded lattice parameters $a = 15.248$ (2) Å, $b = 3.8520$ (4) Å, $c = 10.157$ (2) Å, and $\beta = 118.41$ (1)° (Y₂Cl₃:⁵ C2/m, $a = 15.144$ (3) Å, $b = 3.825$ (1) Å, $c = 10.077$ (2) Å, $\beta = 118.24$ (2)°). The difference in cell volumes is 10.5 Å³. Similarly, cell data obtained for β -Gd₂Cl₃N, $a = 15.290$ (5) Å, $b = 3.912$ (1) Å, $c = 10.209$ (3) Å, and $\beta = 117.79$ (3)°, give a cell volume (540.2 Å³) which is now only 5.5 Å³ greater than that for Gd₂Cl₃² ($a = 15.231$ (3) Å, $b = 3.895$ (1) Å, $c = 10.175$ (2) Å, $\beta = 117.64$ (1)°).

Crystallography. Suitable crystals of β -Y₂Cl₃N were sealed in thin-walled glass capillaries in a glovebox. Diffraction data were collected at room temperature on a CAD-4 diffractometer using graphite-monochromated Mo K α radiation. Details of the data collection and refinement are given in the supplementary material. An empirical absorption correction was first applied by using the average of six ψ -scans at different values of θ , each being the average of Friedel pair data. The structure was solved (SDP program) in space group C2/m starting with the positional parameters of Y₂Cl₃. Successive cycles of least-squares refinement and Fourier map synthesis were used to locate the nitrogen atom, which was found not in the center of the Y₆ octahedra but as an approximately nine-electron residual between the Y₆ octahedra above and below the shared edge in the chain.

At this stage of refinement, the *R* index was ~7% at convergence. Since yttrium lies on the absorption edge of Mo K α radiation ($\mu = 254.1$ cm⁻¹), an empirical absorption correction using DIFABS¹⁷ was applied, and a secondary extinction correction was also made. *R* and *R_w* improved to 3.9 and 4.8%, respectively (Table I). The final difference map was

Table II. Atom Parameters for β -Y₂Cl₃N

atom ^a	x ^b	z	atom ^a	x ^b	z
Y1	0.41182 (4)	0.04978 (6)	Cl2	0.7705 (1)	0.8840 (2)
Y2	0.13503 (4)	0.29197 (6)	Cl3	0.9491 (1)	0.2648 (2)
Cl1	0.6613 (1)	0.5132 (2)	N ^c	0.5790 (6)	0.1639 (8)

^a All atoms in special position 4i with $y = 0$. ^b Standard deviations are in parentheses. ^c Unit occupancy for N.

Table III. Important Distances (Å) in β -Y₂Cl₃N

atom 1-atom 2 ^a	dist	dev ^c from Y ₂ Cl ₃ × 10 ³
Y1-Y1	3.290 (1)	24
Y1-Y2	3.6579 (6)	20
Y1-Y2	3.7290 (7)	35
Y2-Y2	4.4453 (6) ^b	14
Y1-Y1	4.9312 (8) ^b	28
Y1-Cl2	2.7815 (9)	9
Y2-Cl2	2.777 (2)	35
Y2-Cl3	2.713 (2)	14
Y1-Cl3	2.761 (1)	4
Y2-Cl1	2.839 (1)	18
Y2-Cl1	2.776 (1)	1
Y1-Cl2	3.154 (2) ^b	24
Y1-N	2.240 (9)	
Y2-N	2.253 (3)	
N-N	3.042 (9)	

^a All atoms also have two neighbors at the *b* repeat: 3.8535 (4) Å. ^b Distances to adjoining chains. ^c Y₂Cl₃ always has the shorter contact.

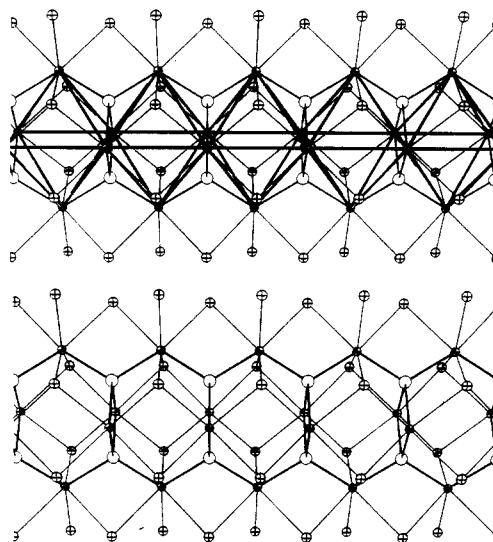


Figure 1. Approximate [201] view of a portion of the infinite chains in β -Y₂Cl₃N (Y, shaded ellipsoids, with Y1 in the shared edge; N, open circles; Cl, crossed ellipsoids, with Cl1 at top and bottom bridging to other chains (50% probability ellipsoids)): (top) emphasis on Y octahedra that share trans edges; (bottom) emphasis on Y-Cl and Y-N bonding. Note that pairs of NY₄ tetrahedra share the Y1-Y1 edge and the other four metal vertices to generate the chains in this representation.

satisfactory with a number of random background residuals of ≤ 1.7 e/Å³. When the nitrogen occupancy and the isotropic displacement parameter were simultaneously varied, the former dropped to 81 (2)% and *B* shrank from 2.9 (2) to 1.8 (1) Å².

Results and Discussion

Positional parameters of β -Y₂Cl₃N determined from a single crystal are given in Table II, and some important distances calculated therefrom are in Table III. Atom displacement parameters and structure factor data are available as supplementary material.

A particularly noteworthy property of β -Y₂Cl₃N is that its powder pattern can be distinguished from that calculated for Y₂Cl₃⁵ only by small line displacements that correspond to increases in the axial lengths of 0.7–0.8%. The compounds are in fact isostructural save for the presence of the weakly scattering nitrogen atom in the former. In addition, the phase contains not

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nitrogen atoms inside each Y₆Cl₈-type cluster, as expected, but rather nitrogen atoms in tetrahedral metal sites between the Y₆ octahedra, above and below the shared Y₆ cluster edges that define infinite chains parallel to the *b* axis. A section of one of these chains is shown in Figure 1 with two emphases: at the top with the metal octahedra in heavy outline and below, with only Y–Cl and Y–N "bonds" highlighted, which is more realistic. The strings of chlorine shown above and below each chain are part of the linkage of the chains at metal vertices into a three-dimensional array, as discussed before.⁵

Nitrogen occupies a special position in the mirror plane that lies normal to the chain and on opposite unit cell faces so that the atom is situated in a distorted Y₄ tetrahedron. The bonds with Y1 in the shared edge show the smallest included angle, 94.5 (2)°, while those with Y2, the vertices of adjacent octahedra, are at an angle of 117.5 (3)°, and the Y1–N–Y2 angles between are 108.9 (3)°. The Y–N distances in β -Y₂Cl₃N (2.241 (8) Å, 2.253 (3) Å) are similar to those observed in (α -)Gd₂Cl₃N (2.262 (6) Å, 2.285 (7) Å) and are consistent with a slightly smaller yttrium, as seen earlier in a comparison of distances in Y₂Cl₃ and Gd₂Cl₃.⁵

The general lengthening of the metal–metal separations on progressing from Y₂Cl₃ to β -Y₂Cl₃N are small, only 0.01–0.04 Å, while changes in *d*(Y–Cl) are only slightly less (Table III), corresponding to an increase of the unit cell volume of only 10.5 Å³ or 2%. The increases in cell lengths and volume in the sequence Gd₂Cl₃– β -Gd₂Cl₃N are in fact only about half as great as those with yttrium. Changes on formation of the yttrium chloride nitride slightly increase the volume of the unoccupied octahedral as well as the occupied tetrahedral sites in the chains.

There are several examples of nitrogen in octahedral interstices in Sc₆Cl₁₂-^{10,11} and Zr₆Cl₁₂-type¹⁸ clusters, and comparisons of those cluster sizes with the cavity available here on the basis of crystal radii differences¹⁹ and with the Y–N separation in YN itself indicate that the alternate octahedral site in β -Y₂Cl₃N is also of a suitable size. Of course, there is the observation that interstitial atoms (other than H) are apparently never found within M₆X₈-type clusters, presumably because of the short distances between interstitial and the face-capping halogens that this would entail.⁸ Clearly, there are also electronic differences that cause twice as much nitrogen to strongly prefer a tetrahedral environment in a valence compound. Factors affecting the stability of alternate phases are obviously complex.

The borderline between a cluster and a valence compound is not as obvious in the case of β -Y₂Cl₃N as it is with the R₃Cl₃/ $(\alpha$ -)R₂Cl₃N pairs, where there is a clear transition from the black sesquichloride to the colorless sesquichloride nitride. The new β -Y₂Cl₃N occurs as black needles that are very fibrous in nature and cannot be cut without fraying, as with Y₂Cl₃. The strength within the chains oriented parallel to the needle (*b*) axis and the relatively lower cohesion between those chains provide a reasonable explanation for the mechanical behavior. As in other interstitial examples, strong yttrium–nitrogen interactions substitute for the yttrium–yttrium bonds in the binary Y₂Cl₃ with the metal–chlorine bonding a common feature in both.

We were not able to find any temperature dependence to the relative yields of α - and β -Y₂Cl₃N, and a normal phase transition between them does not seem likely. Instead, we believe β -Y₂Cl₃N to be somewhat deficient in nitrogen for a valence (Y^{III}) compound, as suggested by the need for a small amount of metal in its synthesis, the refined nitrogen occupancy of 81 (2)%, and the black

color of the crystals. Even though the metal–metal bonding in β -Y₂Cl₃N must be much less important than in Y₂Cl₃, we note that the lowest portion of the occupied metal valence band calculated for Gd₂Cl₃ is largely σ -bonding in the shared edge,¹³ and this may still be partially occupied in a nitrogen-deficient β -Y₂Cl₃N_{1-x}.

The amazingly small changes in dimensions seen on oxidizing Y₂Cl₃ where the average metal oxidation state is +1.5 to the isostructural nitride with an oxidation state of +2.7 (Y₂Cl₃N_{0.8}) to +3 (Y₂Cl₃N) (Table III) clearly highlight the dangers of inferring bond strengths from bond lengths without relevant data; overlap populations that sum over the occupied states involved in bonding these atom pairs are obviously more meaningful. This recalls to mind the many classical examples of short M–M distances that principally derive from M–X packing (matrix effects²⁰) and are unrelated to M–M bonding because of the absence of additional electrons for those states, e.g., in NaCl and CaO. The length of the shared edge Y1–Y1 in Y₂Cl₃, for which the metal–metal bond order is apparently near unity, increases from 3.266 (2) Å only to 3.290 (1) Å in β -Y₂Cl₃N ($\Delta = 11\sigma$), scarcely reflecting what should be the loss of most if not all of the metal–metal bonding electrons. Rather the Y1–Y1 dimer is now bridged on opposite sides by strongly bound nitrogen atoms, and these are presumably responsible for the short but basically *nonbonding* or even repulsive Y–Y contact. A similar geometry pertains to α -Gd₂Cl₃N, where chains of N-centered metal tetrahedra share opposite and very short Gd–Gd edges, which at 3.350 (1) Å are remarkably close to the 3.371 (1) Å⁴ (or 3.349 (1) Å³) lengths of the shared edges between octahedra in Gd₂Cl₃. A strong Coulombic N–N repulsion and very short Cl–Cl contacts along the tetrahedral chain (3.073 Å) have been held responsible. The same N–N separation is found here as well, but Cl–Cl distances along and around the chain are more reasonable, ≥ 3.31 Å, a characteristic feature of this connectivity.⁵ In either case, favorable bonding radii for the closed-shell atoms appear to allow the oxidation of the sesquichloride to take place with minimal changes in dimensions.

Attention regarding interstitial impurities has up to now been focused mainly on the octahedral cluster centers, but we see now that an impurity may also stabilize nominal sesquihalides by occupying tetrahedral sites as well. The possibility remains that sesquihalides like La₂Cl₃,²¹ Y₂Br₃,⁵ Er₂Cl₃, Tm₂Cl₃, and Lu₂Cl₃,²² all once reported to occur in the Gd₂Cl₃ structure, may have also been impurity-stabilized, possibly by nitrogen. In particular, we have been unable to reproduce our earlier report of La₂Cl₃ or to obtain Y₂Br₃ in other than 5–10% yields. Synthesis can be especially challenging when phases like β -Y₂Cl₃N are to be found!

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Supplementary Material Available: Tables of crystal data and anisotropic displacement parameters for β -Y₂Cl₃N (2 pages); a table of observed and calculated structure factor data for the same (5 pages). Ordering information is given on any current masthead page.

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