SYNTHESIS AND STRUCTURE OF TETRAETHYLAMMONIUM OCTACHLORODIRHENATE(III)

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The compound $[(C_2H_5)_4N]_2[Re_2Cl_8]$ (I) has been synthesized by means of the reduction of $[(C_2H_5)_4N]_2[ReO_4]$ by molecular hydrogen under a pressure of ~3 MPa in an autoclave in a medium of concentrated hydrochloric acid at a temperature of 140°C. An x-ray structural investigation of I has been carried out on a CAD4S autodiffractometer [Mo Kα radiation, graphite monochromator. $\omega-2\theta$ scanning, $8° < 2\theta < 50°$, 1938 reflections with $I > l\sigma(l)$]. and the structure has been solved by the direct method and refined by the full-matrix least-squares method to $R_1 = 0.0539$ and $R_w = 0.0568$. The structure of I is characterized by strong crystallographic disordering of the cations and anions, which correlate with one another. The principal interatomic distances in structure I are close to the analogous distances in closely related compounds.

At the present time the most convenient and widely used methods for synthesizing compounds with $[Re_2Cl_8]^{2-}$ anions are: the reduction of perrhenates by molecular hydrogen under pressure in an autoclave in a medium of hydrochloric acid at a temperature of ~300°C [1-7] and the reduction of perrhenates in a hydrochloric acid solution with the aid of hypophosphorous acid [6-10]. The other synthesis methods are generally based on the use of reactions of inaccessible compounds, are more tedious, or have a low yield [6, 7]. According to the data in [3, 4], the reduction of perrhenates in an autoclave under the conditions indicated above is completed with the formation of approximately equimolar quantities of $[ReCl_6]^{2-}$ and $[Re_2Cl_8]^{2-}$. However, it was shown in [11-14] that the maximum reducing power of systems consisting of H_2 (3-5 MPa) and HCl (3-6 M) in the autoclave synthesis of cluster compounds of technetium is observed at a temperature of 180-220°C and that the formation of technetium(W) compounds as the final products under these conditions is apparently stipulated only by the low solubility of these compounds with certain cations. Thus, it was theorized that in the case of rhenium, as in the case of technetium, there is no need to raise the temperature of the autoclave to 300°C or more to obtain octachlorodirhenate anions (as well as other cluster compounds of rhenium) and that it is sufficient to select cations which are such that the solubility of all the intermediate mononuclear reduced species would be sufficiently high and they would not precipitate. The present work was devoted to the experimental verification of this idea,

The selection of cations which can be used in the autoclave synthesis of octachlorodirhenate anions was based on the fact that most tetraethylammonium complex compounds of technetium have a high solubility in concentrated hydrochloric acid solutions [13, 14] and readily undergo reduction in an autoclave to bi- and polynuclear clusters. Therefore, similar behavior on the part of tetraethylammonium compounds of rhenium could be expected. Already the first trial experiments confirmed this hypothesis, and the samples of $[(C_2H_5)_4N]_2[Re_2Cl_8]$ synthesized have been used to synthesize other cluster compounds of rhenium.

For example. the problem of synthesizing a compound with the $[Re_2Cl_8]^{3-}$ anion was posed on the basis of the analogy to technetium. As we know. this problem is of crucial importance for the chemistry of Re-Re multiple bonds [6], since there has not been any structural investigation of d^4-d^5 complexes of rhenium without bridging ligands. In [15] cobaltocene in an acetone solution was used for the one-electron reduction of d^4-d^4 complexes of rhenium. However, d^4-d^5 rhenium complexes suitable for an x-ray structural investigation were not obtained. Therefore, an attempt was made to use ferrocene for this purpose. Unfortunately, the tiny acicular crystals presumably of d^4-d^5 complexes of rhenium which formed with a small yield as a result of the reaction of acetone solutions of $[(C_2H_5)_4N]_2[Re_2Cl_8]$ and fetrocene were amorphous to x rays. However. the search for a single crystal of this compound led to the discovery of a single crystal of the original rhenium complex, which, unlike the product formed directly in an autoclave, had suitable characteristics for an x-ray structural investigation. It was decided to carry out an x-ray structural experiment, since the structure of the compound

$[(C_2H_5)_4N]_2[Re_2Cl_8]$ was of special structural interest in connection with the study of the statistical disordering of similar complexes [16]. The results of the investigation of the **ferrocene** derivative will be published separately.

The starting compound for the synthesis of $[(C_2H_5)_4N]_2[Re_2Cl_8](I)$ was tetraethylammonium perrhenate. which was obtained according to a method similar to the method used to obtain $[(C_2H_5)_4N][TcO_4][17]$. A weighed portion of 7-8 g of $[(C_2H_5)_4N][ReO_4]$ was placed in a quartz test tube, 30 ml of concentrated hydrochloric acid were added, and the test tube was closed by means of a quartz cover and placed within a steel autoclave with a working volume equal to 250 ml. Tbe design of the autoclave and the method used to operate it did not differ from those described in [11, 18, 19]. The initial hydrogen pressure was -3 **MPa,** the time needed to establish the **assigned working temperature (130°C) was 2 h, the** holding time at that temperature was 5 **h,** and the cooling time of the autoclave was -12 h. The yield of the turquoise, finely crystalline precipitate, which was dried on **ashless** filter paper in an atmosphere of air, was equal to 50-60% of the theoretical amount. According to x-ray powder diffraction analysis. the precipitate did not contain foreign impurities. After the blue mother solutions (which contained mainly $[Re_2Cl_8]^{2-}$ ions according to the data from optical spectroscopy) were evaporated in a vacuum desiccator and were subsequently employed as the starting compounds for the **synthesis** of additional portions of $[(C_2H_5)_4N]_2[Re_2Cl_8]$, it was possible to increase the total yield of the final product to 80-90%. The crystals of tetraethylammonium octachlorodirhenate(II1) recovered were not suitable for an x-ray structural investigation. A single crystal of I suitable for this purpose was obtained as an admixture during an attempt to synthesize single crystals of **ferrocene-containing** derivatives of rhenium, as described above.

The x-ray structural experiment was performed on a **CAD4S** diffractometer. The details of this experiment and the principal crystallographic data for 1 are as follows:

Formula	$Re_2Cl_8N_2C_{16}H_{40}$
M	916.53
Space group	$(hk0): k = 2n + 1$, $(h0l): h = 2n + 1$,
	$(0kl): l = 2n + 1$
.a. Å	14.027(2)
b, Å	14.108(2)
c, Å	15.162(3)
α, deg	90
β, deg	90
γ, deg	90
V. Å^3	3090.5(15)
Z	4
ρ(calc), g/cm^3	2.029
Dimensions of the crystal, **mm**	0.15 x 0.2 x 0.3
μ(Mo Ka). cm^{-1}	89.02
Transmission coefficient (max. min)	0.9140, 0.6558
Monochromatic radiation	Mo $K\alpha$ ($\lambda = 0.71073$ Å)
Orientational reflections	
number	25
range (26)	$2114 < 2\theta < 30.72$
Temperature, °C	20 ± 1
Scan technique	$\omega-2\theta$
Range for data acquisition, 2θ, deg	$8 < 2\theta < 50$
Number of independent reflections	
total	2621
with $I > lo(l)$	1938
Number of parameters in the **least-squares**	
refinement	181
R_1	0.0539
R_w	0.0568

The structure of the compound was solved by the direct method according to the SHELXS-86 program and refined by the full-matrix least-squares method with the use of the programs of the SDP system. The populations (μ') of the atoms were refined after the anisotropic refinement of all the nonhydrogen atoms: first with alternate fixation of the temperature parameters and the populations and then with simultaneous refinement of the temperature parameters, coordinates, and populations. After this the populations were normalized to unity on the basis of chemical arguments (and the values for the carbon atoms were averaged). The last stage in the refinement of the structure was carried out with fixed values of the populations (μ). The coordinates of the atoms, the temperature parameters, the populations of the atoms, the principal

TABLE 1. Coordinates, Populations, and Equivalent Temperature Parameters of the Atoms in Structure I

Atom	μ'	μ	x	y	z	B_{eq}, Å²
Re(1)	0,665(1)	0,671	0,03532(4)	0,56908(3)	0,48842(3)	4,23(1)
Re(2)	0,167(1)	0.169	0,0701(2)	0.4694(2)	0,5173(2)	4,30(4)
Re(3)	0,159(1)	0,160	-0,0116(2)	0,5207(1)	0,5694(2)	4,16(5)
Cl(1)	1	1	-0,0330(2)	0,6172(2)	0,3547(2)	5,84(6)
Cl(2)	1	1	-0,0637(2)	0,6771(2)	0.5574(2)	6,31(6)
Cl(3)	1	1	0,1353(2)	0,5858(2)	0,6099(2)	6,08(6)
Cl(4)	1	1	0,1708(2)	0,5301(2)	0,4079(2)	5,75(5)
N(1)	1	1	0,6171(6)	0,604,(5)	0,3616(5)	5,7(2)
C(1)	0,66(2)	0,66	0,530(1)	0,612(1)	0,301(1)	6,2(4)
C(2)	1	1	0.4346(9)	0,602(1)	0,3417(9)	9,7(4)
C(3)	0,73(2)	0,66	0,610(1)	0,684(1)	0,433(1)	8,8(5)
C(4)	1	1	0,699(1)	0,694(I)	0,4902(8)	11,7(4)
C(5)	0,71(2)	0,66	0,620(1)	0,510(1)	0,413(1)	7,0(4)
C(6)	1	1	0,607(1)	0.422 1(7)	0,356(1)	10,8(4)
C(7)	0,72(2)	0,66	0,712(1)	0,610(1)	0.303(1)	7,0(4)
C(8)	1	1	0,7269(8)	0,7010(9)	0,2561(8)	9,4(4)
C(11)	0,34(2)	0,34	0,511(3)	0,622(2)	0,402(2)	8.2(9)
C(13)	0,36(2)	0,34	0,686(2)	0.609(3)	0,443(2)	9(1)
C(15)	0,38(2)	0.34	0,628(3)	0.500(3)	0,314(3)	17(1)
C(17)	0,36(2)	0,34	0,630(4)	0,686(2)	0,289(2)	13(1)

interatomic distances, and the bond angles are presented in Tables 1 and 2, and the anions and cations in structure I are depicted in Fig. 1.

Compound I is built up from tetraethylammonium cations and centrosymmetric $[Re_2Cl_8]^{2-}$ anions, and the principal bond lengths and angles differ only slightly from the analogous characteristics in closely related compounds [6, 20]. However, the crystal structure of this compound is characterized by an unusual type of crystallographic disordering of the cations and anions (Fig. 1). It is seen from the results presented (Table 2) that this occurs owing to the proximity of the shape of the $[Re_2Cl_8]^{2-}$ anions to a regular cube with the chlorine atoms at the vertices, as well as owing to the mobility of the carbon chains in the tetraethylammonium cations. It should be noted that the populations of the different crystallographic positions of the anion ($\mu_{Re(1)} = 0.671$, $\mu_{Re(2)} = 0.169$. $\mu_{Re(3)} = 0.160$) correlate well with the populations of the crystallographic positions of the cation ($\mu_{C(1, 3, 5, 7)} = 0.66$, $\mu_{C(11, 13, 15, 17)} = 0.34$). and it is natural to assume that the variant of the cations with C(1, 3, 5, 7) atoms corresponds to the occupied Re(1) position and that the cations with C(11, 13, 15, 17) atoms correspond to Re(2) and Re(3) positions.

In order to account for these effects, the values of the intermolecular contacts of the cations and anions (Table 2) should be examined in greater detail. It is seen from the data presented that most of the intermolecular contacts of the Cl...C type fall within the range between 3.64 and 4.0 **A.** which corresponds to the normal length of these contacts in such compounds [21]. However, there are two shortened contacts, viz., Cl(4)...C(15) and Cl(2)...C(4), the latter corresponding to a contact between a chlorine atom and an outer carbon atom, which is not crystallographically disordered. Thus, in the centrosymmetric cube of chlorine atoms we can single out four atoms, two of which [Cl(2) and Cl(2)'] always have shortened contacts, while the other two [Cl(4) and Cl(4)'] have shortened contacts only in cases in which the Re(2) or Re(3) position is occupied. The three orientations of the anion are equivalent with respect to the Cl(2) and Cl(2)' atoms, and when the contacts formed by the Cl(4) and Cl(4) atoms with the C(15) atom are taken into account, the inequivalence between the Re(1) atom and the Re(2) and Re(3) atoms becomes evident, but the Re(2) and Re(3) atoms remain equivalent. We note that the shortening of the Cl...C contacts indicated above should be accompanied by significant distortion of the $[Re_2Cl_8]^{2-}$ anions [the mean-square length of the Re—Cl bonds in the $[Re_2Cl_8]^{2-}$ anions calculated on the basis of the literature data [22] is 2.333(12) Å] in all three of its orientations due to the shortening of the Re(1)-Cl(2) bonds or the lengthening of the Re(2)—Cl(2) and Re(3)—Cl(4) bonds. Since in the ideal case of the shape of the $[Re_2Cl_8]^{2-}$ anion in the form of a regular cube, all three orientations of the anion would be equivalent, everything which was stated above allows us to conclude that the $[Re_2Cl_8]^{2-}$ anion avoids the orientation in which the Re atoms are located in a single plane with four Cl atoms having shortened contacts with carbon atoms to the greatest extent. The detailed mechanism of this phenomenon, however, is unclear.

The type of structural disordering under consideration was first described for rhenium compounds in the case of $[(C_4H_9)_4N]_2[Re_2Cl_8]$ [23]. Similar structural disordering was also discovered in other binuclear complexes of rhenium [16, 22, 24-26], molybdenum [27], and technetium [21]. Disordering of the structure in compounds with mobile tetrabutylammonium cations has been noted in mononuclear [17, 28, 29] and polynuclear complexes of technetium [30]. We note that triaxial disordering of the anions {similar to that observed in the case of $[(C_4H_9)_4N]_2[Re_2Cl_8]$} was found only in the structures of the compounds $[(DMFA)_2H]_2[Re_2Br_8]$ [22], $[(C_4H_9)_4N]_2[Re_2I_8]$ [24], and $[(C_4H_9)_4N]_2[TcCl_6]$ [21], equally probable populating of the three crystallographic positions of the anions being observed in the iodide complex and possibly in the technetium complex. The effects of crystalline disordering were examined most thoroughly in [16], but the data from the present work introduce

Bond	d, Å	Bond	d, Å
Re(1)−Re(1)′	2.2146(7)	Re(I)-Cl(1)	2,344 (3)
Re(1)−Cl(2)	2,312(3)	Re(1)−Cl(3)	2.327 (3)
He(1)−Cl(4)	2,324 (2)	Re(2)−Re(2)′	2.21 I (3)
He(2)−Cl(1)	2,352 (3)	Re(2)−Cl(2)	2,357 (3)
Re(2)−Cl(3)	2.347 (3)	Re(2)-Cl(4)	2,341(3)
Re(3)−Re(3)′	2.208 (3)	Re(3)−Cl(1)	2,347(3)
Re(3)−Cl(2)	2.331(3)	He(3)-Cl(3)	2,338(3)
Re(3)−Cl(4)	2.370(3)	N(1)-C(I)	1.53(2)
N(1)-C(3)	1,56(2)	N(I)-C(5)	1.54(2)
N(1)-C(7)	1.59(2)	N(1)−C(11)	1,63(4)
N(1)−C(13)	1.57(4)	N(1)−C(15)	1,65(4)
N(1)−C(17)	1,60(3)	C(I)-C(2)	1,48(2)
C(2)-C(11)	1,43(3)	C(3)-C(4)	1,53(2)
C(4)−C(13)	1,40(4)	C(5)−C(6)	1,52(2)
C(G)−C(15)	1.30(4)	C(i)-C(H)	1,49(2)
C(8)−C(17)	1,46(5)	Cl(1)−C(1)	4,00(2)
Cl(4)−C(2)	3.99(2)	Cl(1)−C(3)	3,64(2)
Cl(1)−C(6)	3,79(2)	Cl(1)-C(7)	3,67(2)
Cl(1)−C(8)	3,87(1)	Cl(1)−C(11)	3,79(3)
Cl(1)−C(15)	3.79(4)	Cl(1)−C(17)	3,73(4)
Cl(2)−C(3)	3.65(2)	Cl(')−C(4)	3.49(2)
Cl(2)−C(6)	3,75(1)	Cl(2)-C(X)	3.80(I)
Cl(2)−C(11)	3,83(3)	Cl(2)−C(17)	3,64(3)
Cl(3)−C(1)	3,73(1)	Cl(3)-c(2)	3,66(1)
Cl(3)−C(4)	3,71(1)	Cl(3)−C(5)	3,70(2)
Cl(3)−C(6)	3,65(2)	Cl(3)-C(i)	3,74(2)
Cl(3)−C(13)	3,81(4)	Cl(3)−C(15)	3,72(5)
Cl(4)-C(2)	3.97(I)	Cl(4)−C(4)	3,96(1)
Cl(4)−C(7)	3,81(2)	Cl(4)−C(13)	3,60(4)
Cl(4)−C(15)	3,45(4)		

Angle	ω, deg	Angle	ω, deg
Re(1)′Re(1)Cl(1)	102,09(6)	Re(1)′Re(1)Cl(2)	103,86(7)
Re(1)′Re(1)Cl(3)	103,48(6)	Re(1)′Re(1)Cl(4)	103,94(7)
Cl(1)Re(1)Cl(2)	87,40(9)	Cl(1)Re(1)Cl(3)	154,42(9)
Cl(1)Re(1)Cl(4)	87,05(9)	Cl(2)Re(1)Cl(3)	86,41(9)
Cl(2)Re(1)Cl(4)	152.20(9)	Cl(3)Re(1)Cl(4)	86,95(9)
Re(2)′Re(2)Cl(1)	101,6(1)	Re(2)′Re(2)Cl(2)	101.2(1)
Re(2)′Re(2)Cl(3)	102,4(1)	Re(2)′Re(2)Cl(4)	103,0(1)
Cl(1)Re(2)Cl(2)	86,1(1)	Cl(1)Re(2)Cl(3)	87,5(1)
Cl(1)Re(2)Cl(4)	155,3(1)	Cl(2)Re(2)Cl(3)	156,3(1)
Cl(2)Re(2)Cl(4)	90.2(I)	Cl(3)Re(2)Cl(4)	86,1(1)
Re(3)′Re(3)Cl(1)	102.1(I)	Re(3)′Re(3)Cl(2)	102,9(1)
Re(3)′Re(3)Cl(3)	103,0(1)	Re(3)′Re(3)Cl(4)	101,4(1)
Cl(1)Re(3)Cl(2)	155,1(1)	Cl(1)Re(3)Cl(3)	87,8(1)
Cl(1)Re(3)Cl(4)	85,9(1)	Cl(2)He(3)Cl(3)	85,7(1)
Cl(2)Re(3)Cl(4)	90,1(1)	Cl(3)He(3)Cl(4)	155,6(1)
C(1)N(1)C(3)	108(1)	C(1)N(1)C(5)	113(1)
C(1)N(1)C(7)	109,2(9)	C(3)N(1)C(5)	105,8(9)
C(3)N(1)C(7)	114(1)	C(5)N(1)C(7)	107(1)
C(11)N(1)C(13)	105(2)	C(11)N(1)C(15)	113(2)
C(11)N(1)C(17)	105(2)	C(13)N(1)C(15)	109(2)
C(13)N(1)C(17)	116(2)	C(15)N(1)C(17)	110(2)
N(1)C(1)C(2)	118(1)	N(1)C(3)C(4)	114(1)
N(1)C(5)C(6)	115(1)	N(1)C(7)C(8)	115(1)
N(1)C(11)C(2)	114(2)	N(1)C(13)C(4)	121(3)
N(1)C(15)C(6)	121(3)	N(1)C(17)C(8)	116(3)

even greater uncertainty into the conclusions regarding the causes and laws of these effects, indicating that further study of this phenomenon is needed.

Thus, although an analysis of the available results does not make it possible to draw unequivocal conclusions regarding the laws and causes of the observed crystallographic disordering of the type under consideration at the present time, it may be stated that a great tendency for it is apparently displayed by compounds with large (relative to the dimensions of the anion) mobile cations, which tend to achieve the most compact type of packing in the crystal cell, as well as anions which simultaneously have a ligand environment with high symmetry and some internal substructure (for example, the pseudocubic $Re_2Cl_8^{n-}$ anion). At the same time, it was noted in [30] that pseudo-octahedral $[Tc_6(\mu_3-Cl)_6Cl_6]^{2-}$ cluster anions are capable of suppressing the statistical disordering of even such mobile cations as $[(C_4H_9)_4N]^+$. This is attributed to the fact that the packing of such very bulky anions in space may result in the formation of very large voids, whose dimensions may be sufficient for accommodating even such bulky cations as tetrabutylammonium ions.
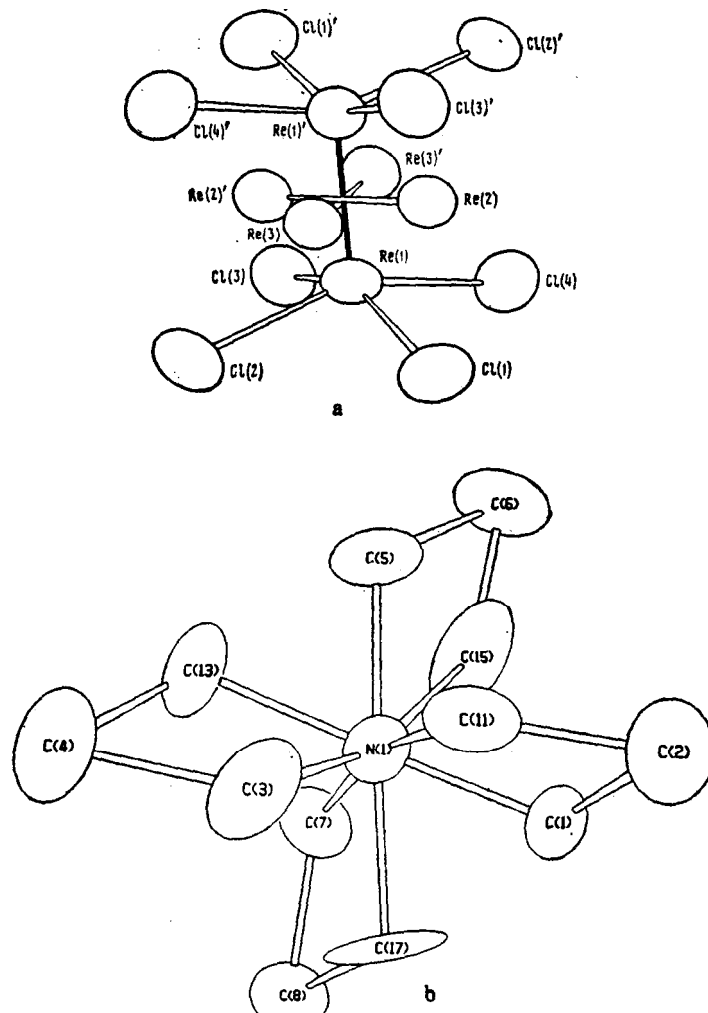
Fig. 1. Diagrams of the statistical disordering of the $[Re_2Cl_8]^{2-}$ anions (a) and the $[(C_2H_5)_4N]^+$ cations (b) in structure I.

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