

The formation of explosive chlorine–nitrogen compounds during the processes of precious metals separation

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Abstract

The formation of the explosive compound NCl_3 during the reaction of ammonium compounds in hydrochloric acid with H_2O_2 or chlorine as an oxidant was investigated. With the use of H_2O_2 in the acidity range from 3 to 6 M only traces of NCl_3 were formed. On the other hand, when chlorine was added to 0.01–2 M NH_4Cl solutions, considerable quantities of NCl_3 were formed by a rapid reaction. At acidities ≤ 3.5 M the reaction yield was 30–65% relative to the chlorine quantity used, depending upon the intensity of the interaction between the solution and the chlorine phases. At acidities > 3.5 M and with rising reaction temperature (20–90°C), NCl_3 formation decreased.

The $\text{NCl}_3/\text{NH}_4^+$ mixtures were metastable. The decrease of NCl_3 concentration in the reaction solution after the chlorine flow had been shut off was mainly due to evaporation of the NCl_3 and only to a small extent due to conversion into the stable final products (N_2 or NO_3^-).

In addition, the formation of NCl_3 in recycling of precious metals (PGM), especially during the precipitation of $(\text{NH}_4)_2[\text{IrCl}_6]$ and $(\text{NH}_4)_2[\text{PdCl}_6]$ and during the conversion of $(\text{NH}_4)_2[\text{PdCl}_6]$ into Pd(II) compounds, was studied. During the precipitation of $(\text{NH}_4)_2[\text{PdCl}_6]$ considerable quantities of NCl_3 were formed. © 1997 Elsevier Science B.V.

Keywords: Explosive compound; Halogen amine; Nitrogen trichloride (NCl_3); Formation; Analysis; Chlorine; H_2O_2 ; Ammonium compound; Pd compound; Ir compound; Acidic solution; Precipitation

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1. Introduction

During the extraction and separation of platinum-group metals, as well as during the preparation of their compounds, redox reactions often proceed in the presence of inorganic and organic amines [1–3]. In this connection there arises the question whether the easy oxidizability of these amines may be a danger. However, we have not found any information in the literature on any explosive reactions in aqueous systems. But the oxidation reactions when using halogen-containing oxidants are an exception. Here, during the conversion into the stable final products (N_2 and nitrate), halogen amines of the composition $NH_{(3-n)}X_n$ ($n = 0-3$) may be formed as intermediate compounds, with trihalides being especially explosive [4]. Nitrogen trichloride (NCl_3), which forms during the reactions with chlorine-containing oxidants, is a very unstable substance: only a very small energy input is required to initiate an explosion in both the liquid and the gas phase. The following systems are considered to be dangerous [5,6]:

- amounts of ≥ 0.5 ml (corresponding to about 0.8 g) of liquid NCl_3 ;
- mixtures with ≥ 10 vol.% of NCl_3 in inert solvents, e.g. chlorinated hydrocarbons;
- mixtures with ≥ 0.5 vol.% [5] or 5–6 vol.% [6] of NCl_3 in the gas phase;
- aqueous solutions with NCl_3 contents ≥ 2000 ppm, corresponding to about 17 mmol l^{-1} NCl_3 , because then the solubility of the NCl_3 is exceeded and a pure NCl_3 phase may be formed. If the amount of NCl_3 is greater than 0.5 ml, one has to expect danger to be imminent (see above).

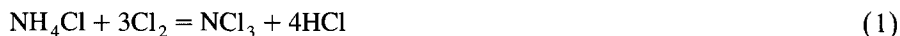
The literature data on the formation of NCl_3 are dispersed and, in part, contradictory (see the summary in [5]).

NCl_3 is formed by the reaction of organic and inorganic amines with chlorine or hypochlorite, but perhaps not when other chlorine-containing oxidants, such as ClO_2 , chlorates or $NOCl$, are used. The reactivity of various compounds can be arranged approximately in the following order [7]:

urea > ammoniumchlorides, sulphate, nitrate \gg acetamide > polyacrylamide
 \gg hydroxylamine.

Complexly bonded amines may also be converted into chloramines by the use of chlorine. For example, by the reaction of Pt(II) or Pt(IV) amines such as Pt(IV)bipyridilamine, complexes with NCl_2 ligands were obtained which, in part, were explosive [8,9].

As to the reaction of ammonium compounds (primarily NH_4Cl) with hypochlorite/chlorine according to the equation



detailed studies for 10^{-4} – 10^{-3} M ammonium-containing solutions within the pH range 1–10 [10–12] and for 0.055–1 M NH_4Cl solutions at 60–80°C for the same pH range [5] are available. For the reaction in the more acidic medium, there are only sporadic references [13,14], as there are for the formation of NCl_3 by electrolysis of ammonium compounds (primarily NH_4Cl solutions) within the neutral to clearly acid range (acidity ≤ 3 M) [7,15–17]. From that it can be concluded that NCl_3 is formed by a rapid reaction at a yield between 30 and 70%, with the formation rate of NCl_3 during

electrolysis of ammonium-containing solutions, at comparable conditions, being greater than the formation rate with chlorine. As to the dependence of NCl_3 formation on acidity, it is found that at $\text{pH} \geq 10$ and at $C_{\text{HCl}} > 6 \text{ M}$ no NCl_3 is formed.

Within the range of $\text{pH} 1\text{--}11$ a formation maximum has been detected at $\text{pH} 2\text{--}4$. However, for the range of $C_{\text{HCl}} = 0.1\text{--}6 \text{ M}$ there are no reliable data.

Though NCl_3 in aqueous phase is unstable in the absence of free chlorine, the reaction with water, excessive amine compounds and other chloroamines proceeds slowly owing to kinetic inhibitions. A rapid reaction resulting in stable final products (NH_4^+ , N_2 or nitrate), however, takes place in alkaline solution ($\text{pH} > 10$) or in the presence of 12 M HCl .

Therefore, considering the available information about the formation and properties of NCl_3 (high vapour pressure, boiling temperature 71°C , possible existence of an azeotropic mixture with water at 53°C , low solubility in aqueous solutions) and the evident metastability of aqueous NCl_3 mixtures, we cannot generally exclude the formation of NCl_3 -containing dangerous systems during the reaction of ammonium compounds with Cl_2/OCl^- in neutral and acid media.

As to technically important reactions proceeding with chlorine in solutions of high ammonium salt content and largely varying acidity, sufficient information about the formation and behaviour of NCl_3 was not available either for systems free of noble metals or for those containing noble metals.

2. The investigation

The formation of NCl_3 was investigated for solutions free of noble metals as well as for solutions with Pd and Ir at various acidities, NH_4Cl concentrations and temperatures, with the use of chlorine or H_2O_2 as an oxidant.

2.1. Experimental details

The experiments were performed with the use of $100\text{--}500 \text{ ml}$ of solution in a glass apparatus capable of being thermostatted. For a given time chlorine was bubbled through the reaction solution under intensive agitation with, at the same time, a given air flow being maintained in the gas space. The chlorine stream was determined by the use of a differential manometer after corresponding calibration, and from that the chlorine quantity introduced into the system was calculated. The reaction gases (if necessary after cooling) were passed through a system of washing bottles. The fractions were processed after the completion of chlorination and a downtime of 5 min .

In view of the tendency of NCl_3 to explode, the following precautions were taken: the apparatus was assembled behind double glass shields; a critical phase composition was avoided by permanently agitating the reaction solution and passing an air flow through the free reaction space.

2.2. Analysis

The quantity of NCl_3 passing from the reaction solution into the gas phase during the experiment, as well as the NCl_3 content in the reaction solution after the experiment was over, were determined. The total NCl_3 quantity results from both values. This quantity is

given in mmol of NCl_3 and in fractions of the added quantities of NH_4Cl and chlorine according to Eq. (1). In addition, the acidity and the remaining NH_4Cl content in the reaction solution after the separation of NCl_3 were determined. The total of this quantity and of the detected NCl_3 quantity was related to the added NH_4Cl quantity and designated as NH_4^+ balance.

The determination of NCl_3 was based on the selective separation of NCl_3 :

1. from the reaction solution, by extraction with CCl_4 followed by conversion with hydrochloric acid to NH_4Cl ; and
2. from the gas phase, by absorption in 12 M hydrochloric acid (in the presence of free NH_3 by absorption in CCl_4).

The final determination of NH_4^+ was performed photometrically in accordance with a modified instruction by de Vries et al. [13].

The detection limits for NCl_3 were 0.007 mmol in the gas phase and 0.0035 mmol NCl_3 in the reaction solution. The correctness of the value obtained was verified by determining the blank value and by model tests using a 0.9 M NCl_3 solution in CCl_4 . This NCl_3 solution was obtained by reaction of a $(\text{NH}_4)_2\text{SO}_4$ solution with chlorine [13] and characterized by ultraviolet spectrography (the peaks obtained at 267 and 345 nm were in accordance with the literature data [5]).

3. Experiments to study NCl_3 formation

3.1. Reaction of NH_4Cl solutions with H_2O_2

In 0.5 M NH_4Cl solutions with an HCl content of 2–6 M, the H_2O_2 content was adjusted to 0.9 M by the addition of 30% H_2O_2 . After a reaction time of 30 min at room temperature as well as at 100°C only traces of NCl_3 were detected (0.005–0.007 mmol NCl_3 , corresponding to $\leq 0.01\%$ of the NH_4Cl quantity added). The NH_4^+ balance was 92–95%; that is, the NH_4Cl was attacked by H_2O_2 only to a negligible degree.

3.2. Reaction of NH_4Cl solutions with chlorine

3.2.1. Dependence of NCl_3 formation upon apparatus conditions

100 and 500 ml of NH_4Cl solution were chlorinated in reaction vessels of different size. The filling levels of the solutions were 80 and 150 mm. The NCl_3 quantities formed at the same specific chlorine flow (given as litres of chlorine per hour and per litre of reaction solution) are shown in Table 1. It can be seen that the NCl_3 yield based on the added chlorine varied between 33 and 62%. Because of the great influence of

Table 1

NCl_3 formation at equal specific chlorine flow. Specific chlorine flow: 15 l $\text{Cl}_2(\text{h})^{-1}$ of solution; chlorination time: 30 min; composition of solution: 0.5 M $\text{NH}_4\text{Cl}/0.5$ M HCl

$V_{\text{solution}}(\text{ml})$	$n_{\text{NH}_4\text{Cl}}(\text{mmol})$	$V_{\text{Cl}_2}(\text{l h}^{-1})$	$n_{\text{Cl}_2}(\text{mmol})$	NCl_3 formation ^a			NH_4 balance(%)
				(mmol)	% Cl_2	% NH_4Cl	
100	50	1.5	34	3.7	33	7.4	97
500	250	7.6	172	35.7	62	14	86

^aThe quantity of NCl_3 formed relative to the expected quantity based on the added quantity of Cl_2 or NH_4Cl .

Table 2

Dependence of NCl_3 formation upon the chlorination rate. Used quantity: 500 ml of 0.5 M NH_4Cl /0.5 M HCl; chlorination time: 30 min

No.	V_{Cl_2}		Cl_2 quantity (mmol)	NCl_3 formation ^a (mmol)	NH_4^+ balance(%)		
	(l h^{-1})	(l (h l)^{-1})			% Cl_2	% NH_4Cl	
1	1.1	2.2	25	5.2	62	2.1	90
2	2.5	5	57	10.3	54	4.1	92
3	2.9	5.8	65	13.9	64	5.6	89
4	5	10	113	24.5	65	9.8	90
5	7.6	15.2	172	35.7	62	14.3	86

^aFor explanation see Table 1.

apparatus conditions on NCl_3 formation, the dependence upon relevant experimental parameters was always determined under the same apparatus conditions.

3.2.2. Dependence of NCl_3 formation upon chlorination rate

With the use of 100 and 500 ml of NH_4Cl solutions the chlorination rate varied. The results are given in Tables 2 and 3. The rise of acidity in the reaction solution was about 110% of the rise that was expected according to Eq. (1).

From the results, it follows that the formation of NCl_3 increased approximately proportionally with increasing chlorine addition.

3.2.3. Dependence of NCl_3 formation upon acidity of solution

Under constant conditions (using each time 0.1 l of 0.5 M NH_4Cl solution of varying acidity, reaction temperature 20°C , chlorination time 30 min, chlorine flow $8\text{--}9 \text{ l h}^{-1}$, chlorine quantity added 180–206 mmol corresponding to 120–137% of the theoretical value for the complete conversion of the added NH_4Cl quantity), the dependence of NCl_3 formation upon the acidity was determined within the range of pH 4.6 to 6.8. The results are shown in Fig. 1, from which the following can be seen:

- The formation rate of NCl_3 was relatively constant within the range of pH 4.6 to about 3.7. The mean value was 19 ± 2 mmol NCl_3 , corresponding to $39 \pm 4\%$ relative to the added NH_4Cl quantity. The NCl_3 contents in the reaction solution were in the range of $21\text{--}30 \text{ mmol l}^{-1}$.
- At acidities > 3.7 M the NCl_3 formation distinctly decreased.

Table 3

Dependence of NCl_3 formation upon the chlorination rate. Quantity used: 100 ml of 0.5 M NH_4Cl /0.5 M HCl; chlorination time: 30 min

No.	V_{Cl_2}		Cl_2 quantity(mmol)	NCl_3 formation ^a (mmol)	NH_4^+ balance(%)		
	(l h^{-1})	(l (h l)^{-1})			% Cl_2	% NH_4Cl	
1	1.5	15	34	3.7	33	7.4	97
2	4.4	44	100	13.1	39	26	91
3	8.5	85	194	20.5	32	41	84

^aFor explanation see Table 1.

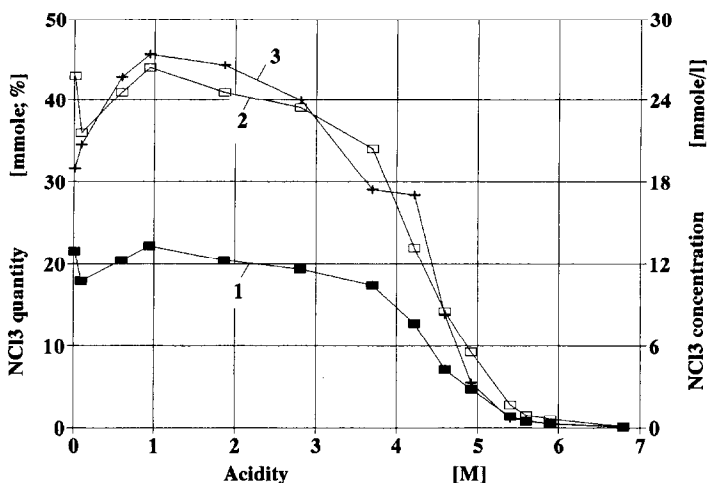


Fig. 1. Dependence of NCl_3 formation upon acidity of reaction solution. Curve 1: NCl_3 quantity formed (mmol); curve 2: NCl_3 quantity formed relative to the NH_4Cl quantity used (%); curve 3: NCl_3 concentration in the reaction solution (mmol l^{-1}).

- From the NH_4^+ balance, it follows that the formation of NCl_3 was the dominant decomposition reaction of NH_4^+ .

3.2.4. Influence of NH_4^+ concentration

Under constant conditions (using 0.1 l of 0.5 M HCl solution, reaction temperature 20°C , chlorination time 30 min, chlorine flow 8.5 l h^{-1} , chlorine quantity added 185 mmol) the NH_4Cl concentration was varied from 0.01 to 2 M.

The results are shown in Fig. 2. The formed quantity of NCl_3 tended to a saturation value corresponding to an NCl_3 concentration in the reaction solution of about 25 mmol l^{-1} .

3.2.5. Influence of reaction temperature

Under constant conditions (using 0.1 l of 0.5 M NH_4Cl solution in 1.97 M HCl, chlorination time 30 min) at two different chlorination rates, the reaction temperature was varied thus:

Series A: $5 \text{ l h}^{-1} \text{ Cl}_2$; chlorine quantity 113 mmol, corresponding to 73% of the theoretical value for the complete conversion according to Eq. (1);

Series B: $9.0 \text{ l h}^{-1} \text{ Cl}_2$; chlorine quantity 203 mmol, corresponding to 132% of the theoretical value.

The results are shown in Fig. 3. The formation of NCl_3 was small at temperatures $\geq 60^\circ\text{C}$, also when a greater chlorine excess was used. The NH_4^+ balance decreased from values $\geq 90\%$ down to 65–70%. This shows that, with rising temperature, competitive decomposition reactions of the NH_4^+ ion become more important.

3.3. Time dependence of the formation and decomposition reaction of NCl_3

With the use of 0.5 M NH_4Cl in solution with 0.5 M hydrochloric acid, the formation rate of NCl_3 was studied for a period of 45 min at a specific chlorination rate of 5.4

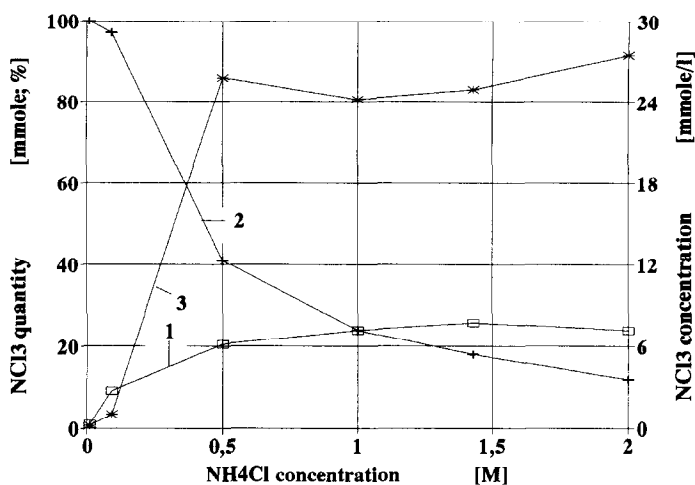


Fig. 2. Dependence of NCl_3 formation upon NH_4Cl concentration in the reaction solution. Curve 1: NCl_3 quantity formed (mmol); curve 2: NCl_3 quantity formed relative to the NH_4Cl quantity used (%); curve 3: NCl_3 concentration in the reaction solution (mmol l^{-1}).

$l(\text{h})^{-1}$. Samples were taken and analysed for both the reaction solution and the off-gas absorption solution. After the chlorine flow had been shut off, agitation of the chlorine-containing reaction solution and flow of carrier gas through the reaction space were continued. Sampling and analyses were done by the method already described. The results are given in Table 4 and are summarized as follows:

- In the reaction solution, a rapid formation of NCl_3 took place, with the greater portion of NCl_3 (93–96%) initially remaining in the aqueous solution.

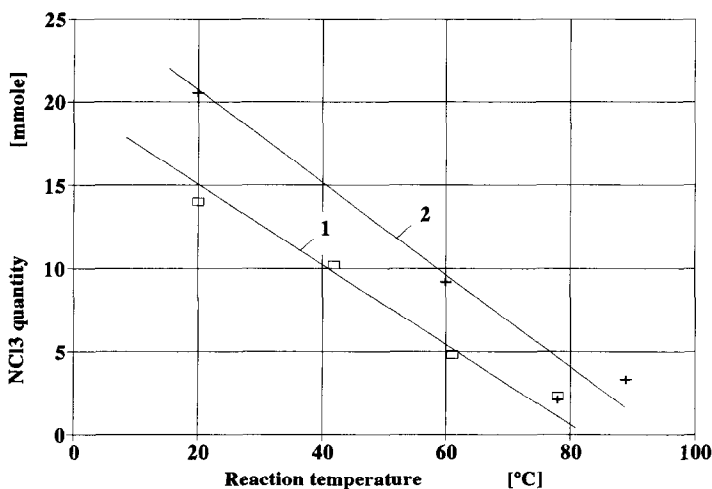


Fig. 3. Dependence of NCl_3 formation upon reaction temperature. Curve 1: chlorine quantity used = 73% of the theoretical value; curve 2: chlorine quantity used = 132% of the theoretical value.

Table 4

Dependence of formation and decomposition reaction of NCl_3 upon time. Quantity used: 0.5 l of 0.5 M $\text{NH}_4\text{Cl}/0.5$ M HCl; specific chlorine flow: 5.4 l(h)^{-1}

Time(min)	Reaction solution		Absorption solution
	C_{NCl_3} (mmol l ⁻¹)	n_{NCl_3} (mmol)	n_{NCl_3} (mmol)
Chlorination reaction			
10	4.4	2.21	0.09
20	12.9	6.33	0.31
30	17.1	8.29	0.67
45	26.4	12.50	2.1
Decomposition reaction			
15	22.9	10.50	— ^a
30	12.9	5.71	— ^a
45	11.4	4.93	— ^a
60	8.1	3.41	— ^a
180	1.57	0.64	10.4
24 h	0.019	0.007	0.1 ^b

^aNo values.

^bThe absorption solution was changed.

• After the chlorine flow had been shut off, the quantity of NCl_3 in the reaction solution decreased by 10.8 mmol within 3 h (allowing for the portion taken off for analysis). Of that, 8.3 mmol = 77% was recovered in the absorption solution.

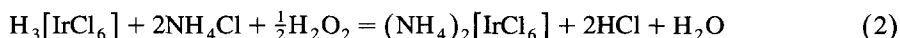
• Of the NCl_3 quantity removed subsequently from the reaction solution during 21 h, which resulted in a decrease of NCl_3 concentration from 1.57 down to 0.019 mmol l⁻¹, only 16% was recovered in the absorption solution.

4. NCl_3 formation in the presence of platinum-group metals

The formation rate of NCl_3 was studied during the precipitation of the ammonium compounds of the Pd(IV) and Ir(IV) chlorocomplexes, proceeding always under oxidizing conditions, and during the conversion of ammonium hexachloropalladate(IV) into Pd(II) compounds.

4.1. NCl_3 formation during precipitation of $(\text{NH}_4)_2[\text{IrCl}_6]$

The precipitation of $(\text{NH}_4)_2[\text{IrCl}_6]$ with the use of H_2O_2 as an oxidant proceeds according to the following equation:



In 0.02 M Ir solutions of different acidity, the NH_4Cl concentrations were adjusted to 0.6–0.9 M and the H_2O_2 content to about 1 M. The $(\text{NH}_4)_2[\text{IrCl}_6]$ was precipitated by heating at boiling temperature for 1 h. The NCl_3 quantities detected are given in Table 5. The formation of NCl_3 during precipitation of Ir with the use of H_2O_2 as an oxidant was very low.

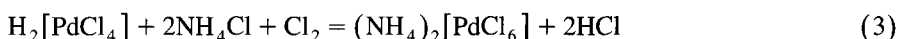
Table 5

Formation of NCl_3 during the precipitation of $(\text{NH}_4)_2\text{IrCl}_6$. Quantity used: 100 ml of 0.02 M Ir solution; oxidant: H_2O_2

No.	Acidity(M)	NCl_3 formation(mmol)
1	4.2	≤ 0.007
2	4.6	≤ 0.007
3	5.0	0.01

4.2. NCl_3 formation during precipitation of $(\text{NH}_4)_2[\text{PdCl}_6]$

The precipitation of the brick-red $(\text{NH}_4)_2[\text{PdCl}_6]$ is performed, in preparative practice, nearly exclusively with the use of excess of chlorine according to the following equation:



In 400 ml of about 0.34 M Pd solution of 4.2 M acidity, the NH_4Cl concentration was adjusted to 1–1.2 M and a chlorine flow of 1.5 h^{-1} was passed through it at room temperature for a period of 4 h. A Pd-free 0.95 M NH_4Cl solution in 4.2 M hydrochloric acid was treated in the same way. The results of NCl_3 determination are given in Table 6. The NCl_3 content in the gas phase was determined, first, when about 80% of the chlorine quantity required for the Pd oxidation had been added (column A) and, second, when the reaction had finished (column B). The NCl_3 content in the reaction solution was determined only at the end of the reaction.

It can be seen that:

- A considerable formation of NCl_3 took place in the used Pd solution of relatively high acidity. Even a distinct chlorine deficiency (column A) did not completely suppress the formation of NCl_3 .
- The NCl_3 quantity formed in the Pd solution was less than in the Pd-free model solution (decreasing to about 30%), which was due to the preferred oxidation of palladium by chlorine.

Table 6

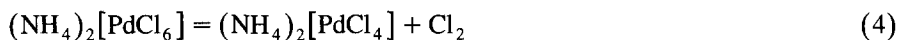
Formation of NCl_3 during the precipitation of $(\text{NH}_4)_2\text{PdCl}_6$

Solution used	NCl_3 quantity formed ^a				
	Gas phase		Reaction solution(mmol)	Total	
	A	B		(mmol)	% Cl_2
Model solution	0.31	8.6	1.5	10.41	14
Pd solution	0.015	2.2	0.73	2.95	3.3

^aFor explanation see Table 1.

4.3. NCl_3 formation during dissolution of $(\text{NH}_4)_2[\text{PdCl}_6]$

During the reaction with aqueous solutions according to the equation



the slightly soluble $(\text{NH}_4)_2[\text{Pd(IV)Cl}_6]$ is converted into readily soluble chloropalladate(II).

The reaction of dissolving was realized by two different variants, each using 20 g of $(\text{NH}_4)_2[\text{PdCl}_6]$:

(a) reaction of an aqueous $(\text{NH}_4)_2[\text{PdCl}_6]$ suspension ($\text{pH} \leq 1$) at 75°C within 30 min, followed by a longer reaction at 60°C after adjustment of the pH value to about 9 by adding NH_3 ;

(b) longer reaction of an ammoniacal $(\text{NH}_4)_2[\text{PdCl}_6]$ suspension ($\text{pH} = 9$) at 60°C .

The NCl_3 quantity formed was about 0.03 mmol in the first variant and below the detection limit of ≤ 0.007 mmol in the second variant.

5. Discussion

5.1. Formation of NCl_3

With the use of H_2O_2 as an oxidant during the reaction with 0.5 M NH_4Cl solution within the acidity range 2–6 M hydrochloric acid, a considerable formation of NCl_3 could not be observed either in systems containing or in those not containing noble metals.

On the other hand, when chlorine gas reacted with NH_4Cl solutions (over an investigated concentration range 0.1–2 M NH_4Cl), a rapid formation of NCl_3 took place. The acidities at which the formation of considerable NCl_3 quantities must be expected ranged from neutral up to a hydrochloric acid concentration of about 5 M. Rising temperatures diminished the formation of NCl_3 ; however, even at about 90°C , noticeable NCl_3 quantities were still formed. The reaction yield of NCl_3 based on the chlorine quantity used was in the range of 30–65%, highly dependent upon the intensity of interaction between the chlorine and the solution phases, so that the absolute values were clearly affected by the apparatus conditions.

For the reactions investigated in the presence of platinum-group metals, a considerable formation of NCl_3 was observed during the precipitation of $(\text{NH}_4)_2[\text{PdCl}_6]$.

5.2. The behaviour of NCl_3 -containing systems

The behaviour of these systems is governed by the noticeable but limited solubility of NCl_3 in aqueous solution, its relatively high vapour pressure, and its remarkable chemical stability against excessive NH_4Cl . Therefore, during the formation stage, the NCl_3 concentration in the reaction solution initially increases, accompanied by a relatively low transition of the NCl_3 into the gas phase. Then, the NCl_3 concentration in the gas phase distinctly increases when the solubility limit of NCl_3 in the aqueous

solution (about 17 mmol l^{-1}) is exceeded. But, because under the conditions chosen the formation rate of NCl_3 is higher than the evaporation rate, NCl_3 contents in the solution may be observed which are distinctly higher than 17 mmol l^{-1} .

When the feed of chlorine has been shut off, the NCl_3 content in the reaction solution decreases considerably within some hours. This is caused mainly by the evaporation of NCl_3 and its removal by the flowing gas phase, whereas the reaction with water or NH_4Cl present in a large excess contributes only a little to it. Reaction with water or NH_4Cl , resulting in stable compounds such as N_2 and NO_3^- , becomes decisive only at low NCl_3 contents.

The NCl_3 can be quantitatively removed from the gas phase by absorption with suitable organic solvents or by reaction with concentrated hydrochloric acid.

From the investigations it follows that during the reaction of NH_4Cl solutions with chlorine the quantity of NCl_3 formed in many cases exceeds the safe limit. This result is important because the application of gaseous or anodically formed chlorine to remove disturbing ammonium compounds from aqueous solutions is generally recommended [18,19]. The risk for safety due to this, which arises in a similar manner when hypochlorite is used [5], either is not known or is underrated.

6. Safety precautions

All the reactions with chlorine and hypochlorite on the one hand and amines on the other must be examined with regard to the possibility of forming chloroamines. In such a case alternatives must be tested in order to avoid this.

If this is not possible, then the formation of NCl_3 must be controlled by measurements and the existence of dangerous conditions must be avoided.

To do this

- the reaction solution must continuously be agitated to avoid the formation of a liquid NCl_3 phase;
- a gas flow must permanently be passed through the reaction space to facilitate the evaporation of NCl_3 and to avoid a critical gas mixture;
- the NCl_3 contained in the gas flows must be removed by suitable washing stages and converted into harmless compounds.

In view of the explosion hazard already caused by small NCl_3 quantities [5], experiments must be carried out only by experienced chemists, taking into consideration sufficient safety precautions.

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