Чернобай

МИНИСТЕРСТВО ОБРАЗОВАНИЯ РЕСПУБЛИКИ САХА (ЯКУТИЯ)

ГОУ «ФИЗИКО-МАТЕМАТИЧЕСКИЙ ФОРУМ
«ЛЕНСКИЙ КРАЙ»»

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XVI МЕЖДУНАРОДНАЯ
ОЛИМПИАДА "ТУЙМААДА"
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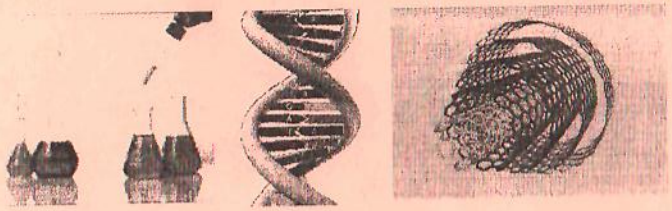
Задания II (практического) тура по химии
The second (experimental) tour on chemistry. Problems



Якутск•Yakutsk•2009

Bromatometric determination of antimony

Bromatometric method is one of the basic approach in redox titrimetry. In this method oxidation by BrO_3^- is used in acidic conditions:

$$BrO_3^- + 6H^+ + 6e \rightarrow Br^- + 3H_2O$$

Potassium bromate $KBrO_3$ is powerful oxidizing reagent ($E^0 = +1{,}45B$), by the way, the rate of the reaction is quite low. So, heating and presence of acid must be the reaction conditions.

During the reaction Br^- ions immediately react with BrO_3^--ions to form bromine, that change color of the solution to pale-yellow:

$$BrO_3^- + 5Br^- + 6H^+ \rightarrow 3Br_2 + 3H_2O$$

In this method acid-base indicators are usually used - like methyl orange or methyl red. Methyl orange oxidation is irreversible process so extra addition of indicator is required.

Bromatometric method is the most useful for arsenic (III) and antimony (III) determination

Determination of antimony (III)

During the titrimetry the following reactions are occurred in the solution:

$$KBrO_3 + 3SbCl_3 + 6HCl \rightarrow KBr + 3SbCl_5 + 3H_2O$$

$$BrO_3^- + 6H^+ + 6e \rightarrow Br^- + 3H_2O$$
$$Sb^{3+} - 2e \rightarrow Sb^{5+}$$

$M_э = 121{,}75/2 = 60{,}88$

Task

1. On your bench you got the test-tubes with HCl ($\omega \approx 15\%$), $SbCl_3$, $BaCl_2$, $ZnSO_4$, NaOH, $Na_2S_2O_3$, $AgNO_3$. Using only this solutions identify each solution. Write chemical equations for each reaction.
2. Determine antimony concentration in the given solution by bromatometric method.
3. Confirm the nature of the antimony acid by chemical reactions.

Reagents: $KBrO_3$ ($c \approx 0{,}1$ M), concentrated HCl ($\rho = 1{,}19$ g/cm^3), methyl orange, distilled water.

Equipment: chemical tripod, burette, vessel, test tubes, pipette, glass, heater.

Method

1. Qualitative detarmination

	HCl	$SbCl_3$	$BaCl_2$	$ZnSO_4$	NaOH	$Na_2S_2O_3$	$AgNO_3$
HCl	-	$H[SbCl_4]$	-	-		$SO_2\uparrow$, $S\downarrow$	$AgCl\downarrow$
$SbCl_3$	$H[SbCl_4]$	-	-	$(SbO)_2SO_4$	$Sb_2O_3\downarrow$ white	$Sb_2O\ S_2\downarrow$ red *****	$AgCl\downarrow$
$BaCl_2$	-	-	-	$BaSO_4\downarrow$	-	$BaS_2O_3\downarrow$ ****	$AgCl\downarrow$
$ZnSO_4$	-	$(SbO)_2SO_4$	$BaSO_4\downarrow$	-	*$Zn(OH)_2\downarrow$ or $Na_2[Zn(OH)_4]$	$BaS_2O_3\downarrow$ ****	$Ag_2SO_4\downarrow$.
NaOH		$Sb_2O_3\downarrow$ *white*	-	*$Zn(OH)_2\downarrow$ or $Na_2[Zn(OH)_4]$	-	-	$Ag_2O\downarrow$ black
$Na_2S_2O_3$	$SO_2\uparrow$, $S\uparrow$	$Sb_2O\ S_2\downarrow$ red *****	$BaS_2O_3\downarrow$****	$Na_2[Zn(S_2O_3)_2]$	-	-	$Ag_2S_2O_3\downarrow$ **,***
$AgNO_3$	$AgCl\downarrow$	$AgCl\downarrow$	$AgCl\downarrow$	$Ag_2SO_4\downarrow$	$Ag_2O\downarrow$ black	$Ag_2S_2O_3\downarrow$ **,***	-

*depends on procedure

**white solid which rapidly becomes black on standing

*** using excess of the reagent $Na_3[Ag(S_2O_3)_2]$ is formed

****during heating precipitate dissolves

***** $SO_2\uparrow$, $S\downarrow$ in excess of acid

2. Bromatometric determination of antimony

A solution of antimony chloride (III) $SbCl_3$ is placed in flask (100 ml), solution of diluted HCl is added. The hydrolysis reaction occurs rapidly but reversible:

$$SbCl_3 + HOH \leftrightarrow SbOCl\downarrow + 2HCl$$

20,00 ml of the solution dissolve to 100 ml in measure flask, then 10-12 ml of concentrated HCl solution is added (d = 1,19 g/cm^3), finally heat till 70°C. After addition of 2-3 drops of methyl red, titrate the solution with $KBrO_3$. At the end of titration add extra more indicator, when colour becomes too pale. Titrate until sharp change in color occurs.

During second titration less amount of $KBrO_3$ (than in the first time) must be added. Heat the solution till 70 °C and after that, add the indicator, and then start titration until formation of colorless solution. .

Calculations

$$M(SbCl_3) = 228\ g/mol \qquad M_3 = 114\ g$$

$$C(1/zX) = (m \cdot 1000)/(M \cdot V)$$

$$m = (C(1/z \cdot X)\ M \cdot V)/1000 \cdot C(1/z\ X) \cdot V(X) = C(1/z \cdot Y) \cdot V(Y)$$

$$C(1/z \cdot X) = (C(1/z \cdot Y) - V(Y)) / V(X)$$

$$T_{A/B} = (C(1/z \cdot A)\ M(B)) / 1000$$

Compounds determination – 7 scores

Chemical equations – 7 scores

Method – 6 scores

Experiment and experimental results – 10 scores

TOTAL: 30 scores