# Determination of hydrazine in water samples

Hydrazine is determined in aqueous samples by redox titration using potassium iodate as a titrant.

| | |
|---|---|
| **Sample** | Hydrazine sulphate, $(NH_2)_2$-$H_2SO_4$<br>0.04-0.09 g |
| **Substance** | $(NH_2)_2$-$H_2SO_4$, M = 130.12 g/mol |
| **Chemicals** | 50 mL deion. water,<br>2 mL conc. hydrochloric acid, HCl (1.16 sg) |
| **Titrant** | Potassium iodate, $KIO_3$<br>c($KIO_3$) = 0.1 mol/L |
| **Standard** | $Na_2S_2O_3$ / KI |
| **Instruments** | DL50Graphix, DL5x, DL7x<br>Analytical balance, printer |
| **Accessories** | Glass titration beaker, ME-101446<br>Rondo 60 Sample changer |
| **Indication** | DM140-SC |
| **Chemistry** | $N_2H_4 + 2H^+ + IO_3^- + Cl^- =$<br>$ICl + N_2 + 3H_2O$ |
| **Calculation** | R1 = VEQ ; mL<br><br>R2 = Q1*C2/m ; %<br>C2 = M/(10*z) |
| **Waste disposal** | Neutralize the aqueous acidic phase before final disposal as anorganic salt solution. |
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## Preparation and Procedures

1.) Weigh 0.04-0.09 g of hydrazine sulphate in a glass titration bealer (polypropylene beakers will be irreversibly stained).

2.) Add 50 mL deionized water.

3.) Add 2 mL concentrated hydrochloric acid (1.16 sg) in order to initiate the desired reaction.

## Remarks

1) The hydrazine level is determined to test the water quality for steam turbine. Hydrazine is used as a scavenger buffer system to ensure greater longevity of those parts of the turbine in contact with the constant steam output.

2) In this application, hydrazine sulphate was directy dissolved in water.

3) According to Vogel's "Textbook of quantitative inorganic analysis" (Ed. 5, 1991), hydrazine can be titrated against potassium iodate releasing iodine monochloride as one of the side-products. The concurrent change in redox states means that a straightforward redox reaction is taking place according to the equation under "Chemistry".

## Results

**METTLER DL70 Titrator** **METTLER TOLEDO UK Ltd**
**Applications Laboratory**

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pgh1 Hydrazine Titration measured 26-Mar-1993 11:08
26-Mar-1993 9:59 Titrator J70820
SW Version 2.0 User Patrick Hogan

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RESULTS

| No | Identification | Weight | | Results | | |
|---|---|---|---|---|---|---|
| 1/1 | (NH2)2S' Aldric | 0.0909 | g | 5.689 | mL | Equival' Vol |
| | | | | 81.423 | % | Hydrazine |
| 1/2 | (NH2)2S' Aldric | 0.0909 | g | 2.936 | mL | Equival' Vol |
| | | | | 79.920 | % | Hydrazine |
| 1/3 | (NH2)2S' Aldric | 0.0909 | g | 3.470 | mL | Equival' Vol |
| | | | | 80.604 | % | Hydrazine |
| 1/4 | (NH2)2S' Aldric | 0.0909 | g | 3.626 | mL | Equival' Vol |
| | | | | 79.947 | % | Hydrazine |
| 1/5 | (NH2)2S' Aldric | 0.0909 | g | 3.554 | mL | Equival' Vol |
| | | | | 81.693 | % | Hydrazine |

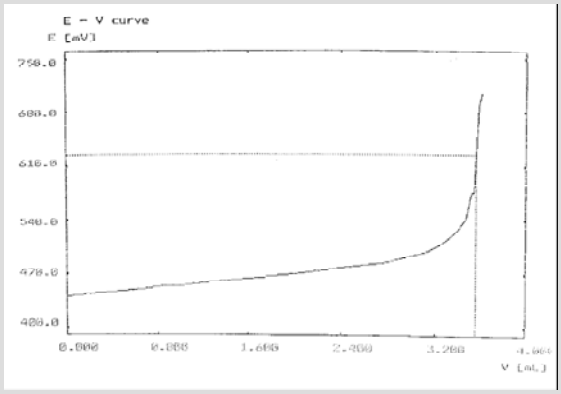
STATISTICS

| | | |
|---|---|---|
| Number results | R2 | n = 5 |
| Mean value | | x = 80.718 % |
| Standard deviation | | s = 0.8205 % |
| Rel. standard deviation | | srel = 1.106 % |

## Table of measured values

| | Volume mL | Increment mL | Signal mV | Change mV | 1st deriv. mV/mL | Time minute |
|---|---|---|---|---|---|---|
| E1 | 0.0000 | | 439.9 | | | 0:00 |
| | 0.5720 | 0.5720 | 448.1 | 8.2 | 14.4 | 0:24 |
| | 0.8580 | 0.2860 | 454.1 | 6.0 | 20.9 | 1:05 |
| E2 | 1.0000 | 0.1420 | 453.7 | -0.4 | -3.1 | 1:35 |
| | 1.2000 | 0.2000 | 457.8 | 4.2 | 20.9 | 2:05 |
| | 1.4000 | 0.2000 | 460.2 | 2.3 | 11.6 | 2:37 |
| | 1.6000 | 0.2000 | 463.6 | 3.5 | 17.4 | 3:05 |
| | 1.8000 | 0.2000 | 466.0 | 2.4 | 11.8 | 3:34 |
| | 2.0000 | 0.2000 | 468.8 | 2.8 | 14.0 | 4:02 |
| | 2.2000 | 0.2000 | 472.8 | 4.0 | 19.9 | 4:28 |
| | 2.4000 | 0.2000 | 476.8 | 4.0 | 20.0 | 4:54 |
| | 2.6000 | 0.2000 | 481.1 | 4.3 | 21.6 | 5:20 |
| | 2.8000 | 0.2000 | 484.9 | 3.8 | 19.0 | 5:48 |
| | 3.0000 | 0.2000 | 492.7 | 7.8 | 39.1 | 6:14 |
| | 3.1340 | 0.1340 | 499.5 | 6.8 | 50.5 | 6:36 |
| | 3.2640 | 0.1300 | 509.3 | 9.9 | 75.9 | 6:56 |
| | 3.3420 | 0.0780 | 518.9 | 9.5 | 122.4 | 7:11 |
| | 3.3900 | 0.0480 | 525.9 | 7.0 | 146.1 | 7:20 |
| | 3.4380 | 0.0480 | 536.1 | 10.2 | 212.3 | 7:29 |
| | 3.4660 | 0.0280 | 541.2 | 5.1 | 183.5 | 7:34 |
| | 3.5180 | 0.0520 | 576.2 | 34.9 | 672.0 | 7:42 |
| | 3.5380 | 0.0200 | 575.4 | -0.8 | -37.8 | 7:45 |
| EGP1 | 3.5580 | 0.0200 | 639.8 | 64.4 | 3221.3 | 7:52 |
| | 3.5780 | 0.0200 | 688.3 | 48.5 | 2424.8 | 8:00 |
| | 3.5980 | 0.0200 | 704.1 | 15.8 | 788.9 | 8:09 |

## Titration curve



## Method

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Method     pgh1      Hydrazine titration
           Version   26-Mar-1993   9:59
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Title
   Method ID  . . . . . . . . . . . . . . pgh1
   Title  . . . . . . . . . . . . . . . . Hydrazine titration
   Date/time  . . . . . . . . . . . . . . 26-Mar-1993   9:59
Sample
   Number samples . . . . . . . . . . . . 1
   Titration stand  . . . . . . . . . . . Stand 1
   Entry type . . . . . . . . . . . . . . Wight m
      Lower limit [mL]  . . . . . . . . . 0
      Upper limit [mL]  . . . . . . . . . 2.0
   ID 1 . . . . . . . . . . . . . . . . . (NH2)2S
   Molar mass M . . . . . . . . . . . . . 130.12
   Equivalent number z  . . . . . . . . . 1
Stir
   Speed [%]  . . . . . . . . . . . . . . 50
   Time [s] . . . . . . . . . . . . . . . 120
Titration
   Titrant  . . . . . . . . . . . . . . . KIO3
   Concentration [mol/L]  . . . . . . . . 0.1
   Sensor   . . . . . . . . . . . . . . . DM140-SC
   Unit of meas.  . . . . . . . . . . . . mV
   Titration mode . . . . . . . . . . . . EQP
      Predispensing . . . . . . . . . . . mL
         Volume [mL]  . . . . . . . . . . 1.0
      Titrant addition  . . . . . . . . . DYN
         dE(set) [mV] . . . . . . . . . . 8.0
         Limits dV  . . . . . . . . . . . Absolute
            dV(min) [mL]  . . . . . . . . 0.02
            dV(max) [mL]  . . . . . . . . 0.2
      Measure mode  . . . . . . . . . . . EQU
         dE [mV]  . . . . . . . . . . . . 0.5
         dt [s]   . . . . . . . . . . . . 1.0
         t(min) [s]   . . . . . . . . . . 3.0
         t(max) [s]   . . . . . . . . . . 30.0
      Threshold . . . . . . . . . . . . . 500.0
      Maximum volume [mL] . . . . . . . . 10.0
      Termination after n EQPs  . . . . . Yes
         n =  . . . . . . . . . . . . . . 1
      Evaluation procedure  . . . . . . . Standard
Calculation
   Result name  . . . . . . . . . . . . . Equival' Vol
   Formula  . . . . . . . . . . . . . . . R1=VEQ1
   Constant . . . . . . . . . . . . . . .
   Result unit  . . . . . . . . . . . . . mL
   Decimal places . . . . . . . . . . . . 3
Calculation
   Result name  . . . . . . . . . . . . . Hydrazine
   Formula  . . . . . . . . . . . . . . . R2=Q1*C2/m
   Constant . . . . . . . . . . . . . . . C2=M/(10*z)
   Result unit  . . . . . . . . . . . . . %
   Decimal places . . . . . . . . . . . . 3
Record
   Output unit  . . . . . . . . . . . . . Printer
   All results  . . . . . . . . . . . . . Yes
   Table of values  . . . . . . . . . . . Yes
   E - V curve  . . . . . . . . . . . . . Yes
   dE/dV - V curve  . . . . . . . . . . . Yes
Statistics
   Ri (i=index) . . . . . . . . . . . . . R2
   Standard deviation s . . . . . . . . . Yes
   Rel. standard deviation srel . . . . . Yes
Rinse
   Auxiliary reagent  . . . . . . . . . . H2O
   Volume [mL]  . . . . . . . . . . . . . 30.0
Conditioning
   Interval . . . . . . . . . . . . . . . 1
   Time [s] . . . . . . . . . . . . . . . 120
   Rinse
      Auxiliary reagent . . . . . . . . . H2O
      Volume [mL] . . . . . . . . . . . . 15.0
Record
   Output unit  . . . . . . . . . . . . . Printer
   All results  . . . . . . . . . . . . . Yes
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