NSWC/WOL/TR 77-71

ANALYSIS OF MERCURIC 5-NITROTETRAZOLE

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RESEARCH AND TECHNOLOGY DEPARTMENT

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Mercury (II) in mercuric 5-nitrotetrazole may be determined by atomic absorption using the unique-to-mercury absorption line at 2536.5 angstroms, with a precision of 1-3%. For a precision of 0.1 to 0.6%, a titration with thiocyanate ion is recommended. The 5-nitrotetrazole anion may be determined by measuring the absorbance due to the anion at 257 nm in the ultraviolet, with a precision of ± 1%. The anion may also be determined by oxidation with dichromate ion in sulfuric acid solution, with a precision

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of ± 0.5%.

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NSWC/WOL/TR 77-71 19 July 1977

ANALYSIS OF MERCURIC 5-NITROTETRAZOLE

This report describes analytical procedures for the determination of the mercury (II) cation, as well as the 5-nitrotetrazole anion, in mercuric 5-nitrotetrazole, in particular for use in a specification. The work was carried out under Task No. WR11HA(OPN).

Julius W. Enig

JULIUS W. ENIG
By direction

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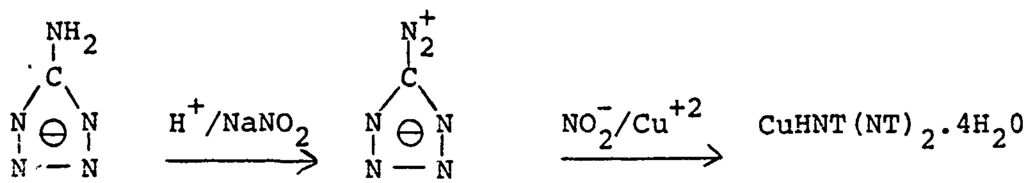
ANALYSIS OF MERCURIC 5-NITROTETRAZOLE

INTRODUCTION

Mercuric 5-nitrotetrazole ($Hg(5\text{-}NT)_2$) is being studied at the NSWC, WOL as a possible replacement for lead azide (1). Unlike lead azide, Hg $(5\text{-}NT)_2$ is stable to moisture and is compatible with detonator construction materials, such as copper or brass.

Twelve batches of Hg $(5\text{-}NT)_2$ ranging from 65 g to 600 g were recently prepared (2) by the procedure of Gilligan and Kamlet (1), according to the following series of reactions:

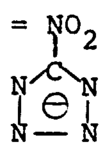


(1)

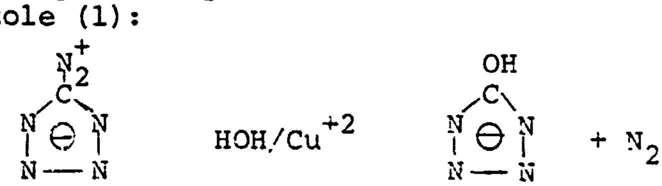
$$CuHNT(NT)_2 \cdot 4H_2O \xrightarrow{CuSO_4/en} Cuen_2(NT)_2 \xrightarrow{HNO_3} Cu(NT)_2 \quad (2)$$

$$Cu(NT)_2 + Hg(NO_3)_2 \longrightarrow Hg(NT)_2 + Cu(NO_3)_2 \quad (3)$$

where NT =



, the nitrotetrazole anion, and en = $H_2NCH_2CH_2NH_2$, ethylenediamine. In addition to these desired reactions, the initial diazo compound (equation (1)) can also react to form the hydroxy-tetrazole (1):



(4)

1. W. H. Gilligan and M. J. Kamlet, "Synthesis of Mercuric 5-nitro-tetrazole", NSWC/WOL/TR 76-146, 9 December 1976.
2. R. E. Farncomb, M. Chang, and F. J. Pisacane, "Process Scale-up for Mercuric 5-nitrotetrazole", personal communication.

It is desirable to have analytical methods for both Hg(II) and the anion, NT, in the final product, as well as for any impurities.

A method is already described (3) for the anion, based on the maximum absorption in the ultraviolet at 257nm. This method is rapid and works well. Another method for the anion, oxidation with dichromate in sulfuric acid, is presented here.

Of the numerous methods in the literature for Hg(II), two were selected for evaluation. A method for determining Hg(II) by atomic absorption (AA) would confirm that this is the element being measured. For this reason, the AA method presented in this report is recommended for specification purposes, even though the method gives a precision of only 1 to 3%. Greater precision may be obtained by the thiocyanate procedure described here.

EXPERIMENTAL AND RESULTS

CAUTION: ANY SALT OF 5-NITROTETRAZOLE SHOULD BE CONSIDERED HAZARDOUS WHEN HANDLED. THE MERCURY (II) SALT HAS THE SAME IMPACT SENSITIVITY, 5 CM., EITHER WET OR DRY (reference 2).

Determination of 5-nitrotetrazole anion

UV Method

Accurately weigh approximately 2.5 mg of Hg $(5\text{-NT})_2$ into a 25 ml volumetric flask. Dissolve in 2.5 ml of 20% ammonium acetate solution, dilute to volume with water, and mix. This is Solution A.

Pipet a two ml aliquot of Solution A into a 10 ml volumetric flask, dilute to volume with water, mix, and read the absorbance at 257nm. Frequently, acetone is used in cleaning volumetric glassware. Even a trace of acetone will interfere at 257 nm and must not be used with any glassware used in this determination.

The ammonium acetate solution is necessary for dissolution of the Hg $(5\text{-NT})_2$ in a reasonable time. Hg $(5\text{-NT})_2$ is soluble in water alone, but slowly, 89.7 mg/100ml dissolving in 3 days at 30°, with 95% of this dissolving in 7 hours.

Using the above procedure with a specially purified sample of Hg $(5\text{-NT})_2$, the molar absorbancy was found to be 5353 ± 28 (average and average deviation for four samples). NMR was used to establish

(3) N. J. Blay, D. G. Davies, D. C. Mullenger, and R. J. Rapley, "Silver Nitrotetrazole: Its Stability and Compatibility with Other Materials", ERDE Technical Report No. 163, January 1974.

the absence of water and any material having hydrogen atoms. A sample of sodium 5-NT was recrystallized from acetone and air dried. If dried in a desiccator, this salt lost its water of hydration (NMR), but if left loosely stoppered on the lab bench, it was shown by NMR to be the di-hydrate. Four samples of this di-hydrate gave 5329 $\pm$ 10 for the molar absorbancy of the NT anion at 257nm.

Fourteen different samples of Hg $(5\text{-NT})_2$ were analyzed by the UV method and the results are shown in Table I.

Dichromate method (4)

Accurately weigh a 35-44 mg sample of Hg $(5\text{-NT})_2$ and transfer it to a 250 ml standard tapered Erlenmeyer flask, using 20 ml of water. Pipet 10 ml of 0.2500 N $K_2Cr_2O_7$ into the flask, then add 30 ml of 96% sulfuric acid, which contains 10 g of Ag_2SO_4/liter. Mix thoroughly. Connect the flask to a reflux condenser and reflux at least 30 minutes. Carefully rinse the condenser with distilled water, and cool the flask to room temperature. Dilute the solution to about 125 ml, again cool, add 2 drops of ferroin indicator and titrate with standardized 0.1 N ferrous ammonium sulfate solution. The end point is very sharp, 0.02 ml, going from green to light brown.

All glassware must be cleaned by using the above procedure without the sample. The distilled water must not be stored in plastic wash bottles. A blank must be run using everything except the sample. The milliequivalents of dichromate consumed are calculated by subtracting the sample titre from the blank titre, and multiplying the difference by the normality of the Fe^{+2}. When the milliequalents thus found are divided by the millimoles of anion (= 2 x millimoles of Hg $(5\text{-NT})_2$), a value of 6 is found, which corresponds to the expected electron change (see Discussion).

The Ag_2SO_4 is used as a catalyst (4), and if it is omitted, the reaction is only 80-90% complete in 30 minutes. With the catalyst, the reaction is complete in 15 minutes.

The results using this procedure are given in Table I, for both Hg $(5\text{-NT})_2$ and Na 5-NT.2HOH.

Determination of Mercury (II) cation

AA Method

Prepare a standard solution of 1000 ppm of Hg(II) by dissolving 1.080 g of mercuric oxide in 5 ml of 6 N nitric acid in a one liter volumetric flask, and diluting to volume with water. Do not use

(4) "Standard Methods for the Examination of Water and Wastewater", edited by H. P. Orland, 12th ed. 1965, p. 510, "Chemical Oxygen Demand".

TABLE I

MERCURIC 5-NITROTETRAZOLE ANION DETERMINATION

Sample (a)	UV Absorption: No. of Detns.	Percent Found	Ave. Dev.	Percent of calcd. (b)	Ave. Dev.	Dichromate Oxidation: No. of Detns.	Meq./Mmole	Ave. Dev.	Percent of calcd. (c)	Ave. Dev.
96-8898-80	5	53.37	0.32	100.3	0.6	10	5.983	0.025	99.7	0.42
Modified	6	51.83	0.21	97.4	0.4	2	6.045	0.015	100.8	0.25
WOL(M)	8	52.62	0.32	98.9	0.6	2	6.034	0.030	100.6	0.50
WOL(P)	6	52.52	0.53	98.7	1.0	4	6.042	0.025	100.7	0.04
Batch 2	7	52.47	0.42	98.6	0.8	2	6.019	0.032	100.3	0.53
" 3	8	52.31	0.42	98.3	0.8	2	6.098	0.026	101.6	0.43
" 4	7	52.84	0.58	99.3	1.1	2	5.990	0.016	99.8	0.27
" 5	6	52.52	0.32	98.7	0.6	2	5.990	0.020	99.8	0.33
" 6	9	47.46	0.28	89.2	0.6	2	5.649	0.028	94.2	0.50
" 7	6	52.47	0.52	98.6	1.0	1	5.975	--	99.6	--
" 9	6	52.73	0.47	99.1	0.9	2	5.964	0.005	99.4	0.08
" 10	5	53.10	0.32	99.8	0.6	2	5.986	0.020	99.8	0.33
" 11	5	52.89	0.32	99.4	0.6	2	6.000	0.019	100.0	0.32
" 12	5	52.78	0.53	99.2	1.0	2	5.930	0.028	98.8	0.47
Na 5-NT.2HOH						4	5.988	0.012	99.8	0.20

(a) The first sample is a laboratory prep.; all others are large scale, 65g to 600g batches; WOL(M) and WOL(P) are either Batch 1 or 8 of a series of 12 batches.

(b) Calcd. for Hg $(5\text{-NT})_2$: anion = 53.21%; Hg = 46.79%.

(c) Based on 6 electron change.

Hg(I) compounds as standards, as they give erroneously high results (5). Dilute aliquots of this standard to give solutions containing 30, 40, and 50 ppm of Hg(II). Read the absorbancy of Solution A above at 2536.5 angstroms and compare with the diluted standards. The machine sensitivity must be adjusted so that the 50 ppm solution reads about three-fourths of the dial. For the Varian Techtron AA-5, this reading is 75 using the times 10 control.

The results using this procedure on Hg $(5\text{-NT})_2$ are given in Table II.

Thiocyanate Method

Pipet an aliquot of Solution A into a suitable titration vessel (a 25 ml volumetric flask may be used), add 1 ml of 6 N nitric acid, 1 Ml of 0.1 N ferric nitrate, mix and titrate with 1×10^{-3} M sodium thiocyanate solution to the light brown end point. Determination of the end point is facilitated by comparison with the color of the starting solution and a solution of the standard Hg(II) that has been titrated. A solution that is 50 ppm in Hg(II) is 2.5×10^{-4} M. The end point occurs when two moles of SCN^- per mole of Hg(II) have been added. Consequently, an aliquot of 8 ml will require 4 ml of SCN^-. A micro buret graduated to 0.01 ml is therefore required.

The results using this procedure are given in Table II.

The end point is easier to detect, if about 50 mg of Hg $(5\text{-NT})_2$ are titrated using 0,1 N SCN^-. The sample is dissolved in 2.5 ml of 20% ammonium acetate, and because ferric ion gives a red color with ammonium ion, 3 ml of 6 N HNO_3 are required to discharge this color. The more dilute solution above was used to avoid unnecessary handling of sensitive Hg $(5\text{-NT})_2$.

Determination of Copper (II) cation

AA Method

Copper (II) is a likely impurity in Hg $(5\text{-NT})_2$, because it is used during the synthesis. A 1 ppm solution of Cu(II) gives a dial reading of 100 on the times 10 dial setting on the Varian Techtron AA-5, whereas 50 ppm Hg(II) gives a dial reading of 75. Therefore, up to 2% Cu(II) in Hg(II) can be determined using the same solution. On this instrument, the Cu and Hg lamps may be mounted at the same time. After determining the absorption due to Hg(II), one changes the lamp by rotating a disk, adjusts the wavelength, and then determines the absorption due to Cu(II).

The results using this procedure are given in Table II.

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(5) D. N. Hingle, G. F. Kirkbright, and T. S. West, Analyst, 92, 759 (1967); Chem. Abstracts, 68, 111,101g (1968).

TABLE II

MERCURIC 5-NITROTETRAZOLE DETERMINATION OF MERCURY AND COPPER

Sample	Atomic Absorption					Thiocyanate Titration			
	Percent Hg(II)(a)				Percent Cu(II)	Percent Hg(II)(b)			
	Found	Ave. Dev.	Percent of calcd.	Ave. Dev.		Found	Ave. Dev.	Percent of calcd.	Ave. Dev.
96-8898-80	47.1	1.1	100.7	2.3	0.012	46.79	0.05	100.0	0.1
Modified	45.2	1.0	96.6	2.2	0.004	46.60	0.28	99.6	0.6
WOL(M)	45.2	0.7	96.6	1.5	0.034	46.70	0.05	99.8	0.1
WOL(P)	45.4	1.1	97.0	2.4	0.038	46.70	0.05	99.8	0.1
Batch 2	47.3	1.0	101.1	2.1	0.028	46.37	0.05	99.1	0.1
" 3	45.4	1.2	97.0	2.6	0.039	46.70	0.09	99.8	0.2
" 4	45.2	0.7	96.6	1.5	0.045	46.65	0.05	99.7	0.1
" 5	44.8	0.7	95.7	1.6	0.037	46.79	0.09	100.0	0.2
" 6	45.9	1.4	98.1	3.1	0.11			(c)	
" 7	46.4	1.1	99.2	2.4	0.045	45.71	0.14	97.7	0.3
" 9	45.8	0.7	97.9	1.5	0.042	46.46	0.19	99.3	0.4
" 10	45.4	0.4	97.0	0.9	0.030	46.42	0.05	99.2	0.1
" 11	45.1	0.7	96.4	1.6	0.032	47.02	0.05	100.5	0.1
" 12	45.3	0.8	96.8	1.8	0.046	46.51	0.09	99.4	0.2

Mercuric oxide plus HNO_3 = reference (see text)

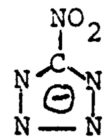
(a) average of two determinations

(b) 96-8898-80, five detns.; Batch 3, three detns.; all others, two detns.

(c) 96.0%, 102.9% (see discussion)

DISCUSSION

For the dichromate oxidation of the 5-nitrotetrazole anion, the equation may be written:



$$+ 3HOH = CO_2 + 2N_2 + NO_3^- + 6H^+ + 6e \qquad (5)$$

Duplicate analyses for nitrate by a gas chromatographic procedure (6) gave 108% ($\pm$ 8%) of the value required by equation (5). Carbon dioxide and nitrogen were not determined. The dichromate oxidation has been used successfully for other explosives in this laboratory (7). The 6 electron change shown above was verified by the experimental results for sample 96-8898-80 of Hg $(5\text{-NT})_2$, and for Na 5-NT. 2HOH (Table I). The value of 6 was invariant for reflux times of 15 minutes to 4 hours.

Comparing the results for the anion procedures in Table I, the values found by the dichromate procedure are generally 0-3% higher than those found by the UV procedure (the results for Batch #6 will be considered separately below). This may be because the dichromate procedure will measure any readily oxidizable material, for example, organic fibers, or Hg(I).

When the samples of Hg $(5\text{-NT})_2$ were received, they were under water. A portion was removed and placed in an open container, which was placed in a desiccator over concentrated sulfuric acid to remove any water. To insure that they were dry, the NMR spectrum in deuterated DMSO was observed. No water was found, nor any hydrogen atoms. Thus, organics, such as ethylenediamine, are absent. It is estimated that the NMR method will detect impurities, if present to greater than 3%.

When the results for mercury (II) by the two procedures given in Table II are compared, and the larger average deviation of the AA method is considered, it is seen that the two procedures agree within 0-2%, except for sample 5 where the difference is 2.5%. Generally, the values by AA are lower than those by thiocyanate. When the AA values are higher, it may mean that mercury (I) is present (samples 2, 6, and 7), although there is no experimental proof of this (5).

(6) D. J. Glover and J. C. Hoffsommer, "Gas Chromatographic Analysis of Nitrate and Nitrite Ions in Microgram Quantities by Conversion to Nitrobenzene", J. Chromatography, 94 334 (1974).

(7) For TNT: $C_7H_5N_3O_6 + 17HOH = 7CO_2 + 3NO_3^- + 39H^+ + 36e$; found, 36.1 $\pm$ 0.3, RDX: $C_3H_6N_6O_6 + 9HOH = 3CO_2 + 3NO_3^- + 3NH_4^+ + 12H^+ + 12e$; found, 11.8 $\pm$ 0.3

Comparing the data in Tables I and II, the values for the anion by UV appear to be greater than the Hg(II) values by AA, but again, when the deviations are considered, the values by these two methods are in agreement. Except for samples 6 and 7, the anion values by dichromate oxidation agree quite well with the Hg(II) values by thiocyanate. Sample 7 may have Hg(I) present as already mentioned.

Sample 6 is the only sample examined which gives different results for Hg and the 5-NT anion by all four procedures. According to reference (2), during the preparation of sample 6, the desired temperature of 75° could not be reached, and this may account for its low purity.

When treated with ammonium acetate solution, or water only, some of sample 6 did not dissolve. When a large sample of 6 (111 mg) was triturated with a solution of 20% ammonium acetate, diluted with water, and filtered, there was found 7.4% of a yellow solid residue. The filtrate from this solid was analysed for the 5-NT anion by UV and for Hg(II) by thiocyanate titration. 87.7% of the theoretical amount of anion was found and 87.0% of Hg(II). When this percentage of Hg $(5\text{-NT})_2$, 87.4%, is added to the solid residue, 94.7% of the sample weight is accounted for. This means that 5.3% of the sample weight in solution is not Hg(II) or 5-NT anion.

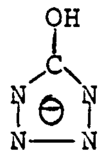
It is possible that the soluble 5.3% is a Hg(I) compound, because the AA analysis for Hg is 98.1%, and Hg(I) compounds give higher absorptions than Hg(II) compounds (5).

It was also considered likely that a soluble nitrate could be the impurity in sample 6. Consequently, a nitrate analysis was done according to (6). There was found 0.48% nitrate, which corresponds to 0.73% $Cu(NO_3)_2$. Converting this value of $Cu(NO_3)_2$ to percent Cu, 0.25%, shows that the nitrate is present not only as the copper salt, for Cu by AA is only 0.11%. Nitrate obviously does not account for the 5.3% soluble in sample 6.

Another possibility for the soluble impurity would be an acidic material. When sample 6 was triturated with water, a pH of 2.99 was found, while the pure sample 96-8898-80 gave a pH of 3.27. When these pH values are converted to H^+ concentrations, there is found for sample 6, 10.23×10^{-4} M, and for 96-8898-80, 5.37×10^{-4} M. The difference, 4.86×10^{-4} M, would be the normality of the acidic impurity. If the acid impurity is hydroxytetrazole (molecular weight = 86), then the weight of hydroxytetrazole is $86 \times 4.86 \times 10^{-4} = 0.0418$ g/liter. As the sample weight was 0.006467 g/10 ml, hydroxytetrazole content would be 6.5%. This value is in fair agreement with the experimental value of 5.3%.

Titration of sample 6 with thiocyanate without filtration of the insoluble material, gave a first end point at 96% of the calculated Hg(II). This end point faded, and a final end point was found at 102.9% of the calculated value. Since the end point did not fade when the insoluble material was filtered, the insoluble material is probably a Hg(II) compound. Considering the value of 102.9%, the molecular weight of the insoluble material must be less than that for Hg $(5\text{-NT})_2$, 428.7 g/mole. It is noted that mercuric 5-hydroxy-tetrazole has a molecular weight of 370.6 g/mole.

The dichromate oxidation of sample 6 without filtration of the insoluble material, gave a value of 94.2% of the sample weight, based on the expected 6 electron change for 5-nitrotetrazole. If, indeed, hydroxytetrazole is present, either as the acid or Hg(II) salt, then the electron change expected is 4:



$$+ \ 3/2HOH = CO_2 + 2N_2 + 3H^+ + 4e \tag{6}$$

This means hydroxytetrazole as an impurity would give low results by the dichromate oxidation. However, these results will be higher than the anion by UV, if the 5-hydroxytetrazole anion absorbs less than the 5-NT anion, which is what is observed (Table I).

SUMMARY AND CONCLUSIONS

Mercury (II) in mercuric 5-nitrotetrazole may be determined by atomic absorption using the unique-to-mercury absorption line at 2536.5 angstroms, with a precision of 1-3%. For a precision of 0.1 to 0.6%, a titration with thiocyanate ion is recommended. The 5-nitrotetrazole anion may be determined by measuring the absorbance due to the anion at 257 nm in the ultraviolet, with a precision of $\pm$ 1%. The anion may also be determined by oxidation with dichromate ion in sulfuric acid solution, with a precision of $\pm$ 0.5%.

For specification purposes, there are advantages to using both of the cation methods presented here, as well as using both anion methods. The AA method for Hg depends on measuring the unique-to-mercury absorption line at 2536.5 angstroms. Hg(I), if present either from Hg(II) contamination, or by reduction of Hg(II), will show up by giving higher than calculated values based on Hg $(5\text{-NT})_2$. The thiocyanate titration gives more precise values for Hg(II), and will react with Hg(I) to give the insoluble salt, and thereby give low results. The UV method for the anion is specific, but is subject to interference by any material absorbing at 257 nm. The dichromate oxidation of the anion will also reflect any easily oxidizable impurity.

Examination of the results for the fourteen different samples of Hg $(5\text{-NT})_2$ presented in Tables I and II, show that one (Batch 6) gives analyses indicating a poor product. On the basis of all the data, the following requirements are recommended:

Property	Requirement
Assay	
Hg (AA)	100 ± 3%
Hg (thiocyanate)	99.5 ± 0.5%
5-nitrotetrazole anion	
UV	100.0 ± 1.0%
Dichromate oxidation	100.0 ± 1.0%
Cu (AA)	<0.05 ± 0.01%
NO_3^- (ref. 6)	<0.10 ± 0.01%
Insoluble in 20% ammonium acetate	0.5% max.
pH (10 mg triturated with 10 ml water)	3.32 ± 0.10

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