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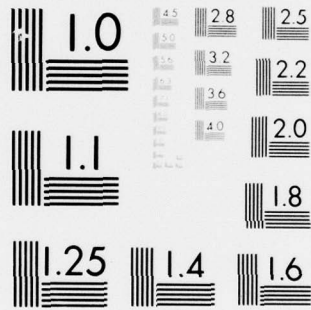
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ACCELERATED STORAGE TRIALS OF TWO COMPONENTS OF THE DETECTOR KIT, CHEMICAL AGENT, C2

by

J.M. Preston and A.A. Casselman



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J.M. Preston and A.A. Casselman
Chemical Defence Section
NBC Defence Division

⑪ May '77

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ABSTRACT

Accelerated storage trials indicate that both the methanol solution of N-ethyl-N-(2-hydroxyethyl)aniline and the "blue-band" tubes, as packaged for the Detector Kit, Chemical Agent, C2 have shelf lives in excess of five years.

RÉSUMÉ

Des essais d'entreposage accéléré indiquent que la solution au méthanol du N-éthyle, N-(hydroxy-2 éthyle) aniline et les tubes "à bande bleue", tels qu'emballés dans la Trousse de detection, Agent chimique, C2, ont une durée de vie en stock de plus de cinq ans.

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INTRODUCTION

This note describes one of the final stages of the development of the Detector Kit, Chemical Agent, C2 (1,2), namely, definitive storage trials of two components. The kit contains two small items of detection equipment and four chemical compounds, all of which must have a shelf life of at least five years. One item, the Detector, Chemical Agent, Nerve Vapour, will be included in the kit in its original packing, in which it has a sufficient lifetime (3). One compound, Chloramine-T, will decompose very slowly since it is a crystalline solid. Sodium hydroxide pellets will be packed in airtight bottles and may show signs of deterioration; however, the solution produced by addition of water will be sufficiently alkaline. The third compound is ethylene glycol, an extremely stable substance. Thus our attention turned to the methanol solution of N-ethyl-N-(2-hydroxyethyl) aniline and the "blue-band" tubes.

The "blue-band" tube is used for detection of all the non-nerve agents in the kit's repertoire. It is a glass tube, 4 mm OD, 2 mm ID, containing a plug of silica gel impregnated with 4-(4'-nitrobenzyl)pyridine (DB3) and mercuric cyanide. The tubes are manufactured by Mine Safety Appliances Co. of Canada Ltd. with sealed ends in which case the storage lifetime is entirely adequate. For use, the ends must be broken off along etchings on the glass, which is a difficult operation when CW gloves are worn and which exposes sharp edges capable of damaging protective equipment. For this kit, therefore, the ends will have been removed and the edges fire-polished before packing. An appropriate packing was found after some experimentation (4) to be lead foil, with seams soldered shut. The storage trials of these packets are described here.

For the detection of phosgene one drop each of base and of N-ethyl-N-(2-hydroxyethyl)aniline are added to the silica gel in the "blue-band" tube, producing a red dye if phosgene has been sampled (5,6). The amine, sealed in glass ampoules to prevent its oxidation, had successfully completed preliminary storage trials (4). However it was decided that these trials should be repeated for several reasons: the ampoules had not been made by a method representative of the commercial process; only neat amine, rather than the methanol solution (used to reduce the viscosity to facilitate dropping onto the silica gel), had been tested; and qualitative analysis of the aged compound had not been performed.

EXPERIMENTALN-Ethyl-N-(2-hydroxyethyl)aniline

N-Ethyl-N-(2-hydroxyethyl)aniline, 50 ml, was purified by fractionation *in vacuo*. The first and last 5% of the distillate were discarded and the middle fraction (b.p. 104-106°C, 0.65 Torr) retained. A 25-ml sample of the distillate was transferred in a dry box to a flask with a ground-glass stopper and 25 ml of a 70% v/v amine/methanol solution prepared in a similar flask using absolute methanol. After retaining small samples of the neat amine and of the solution the flasks were shipped to Canadian Custom Packaging Ltd., Toronto, where they were sealed into glass ampoules (2/3 ml in each) using a commercial instrument. After the chemical was injected, and before the ampoule was sealed, dry nitrogen was injected to replace the air remaining in the ampoule.

The neat amine, shortly after distillation, was shown by GSC (7) to contain approximately 0.5% of an unknown impurity. Other impurity peaks (generally less than 0.05%) were also present but were ignored. The only observable difference between the samples retained at DREO and those returned to us inside the ampoules was that the neat amine had become contaminated with methanol, presumably due to failure to clean the apparatus between runs.

The ampoules were stored in four different ways, at 65°C, at room temperature in the dark and on a window sill, and in a programmed temperature cycle. The initial temperature of this program was 44°C which was increased every Friday by one degree, to 71°C by the end of the 28 weeks. Ampoules were removed from this trial for analysis every four weeks; after 28 weeks the trials were terminated and all samples analyzed.

All analyses were performed by gas-solid chromatography in accordance with a method previously developed here (7). Compound was removed from the ampoules for injection into the GC in three ways, all of which gave identical results. One method (8) was to insert the ampoule into a short piece of Tygon tubing, clamp the ends, break the ampoule, and obtain samples by insertion of a syringe. Another technique was to place the intact ampoule in a Reacti-Vial (Pierce Chemical Corp.) whose cap had been fitted with a rubber septum. The ampoule was then broken with a large needle and sample withdrawn by a syringe. Alternatively the ampoule was scored near one end with a diamond pencil, the end was neatly removed, and the ampoule was then placed in a Reacti-Vial from which samples were withdrawn as before. This was found to be the most appropriate and convenient method. There was no evidence of contamination by the atmosphere although over an extended period the methanol concentration could be observed to decrease slightly.

"Blue-Band" Tubes

Tubes, purchased from Mine Safety Appliances Co. of Canada Ltd., were broken on the etched lines and the ends were fire-polished. From half of the tubes the blue band had previously been removed by soaking in trichloroethylene. Five tubes were packed in each lead foil envelope (foil from Montebello Metal Ltd., Hawkesbury) and the seams were sealed by dipping in molten solder. Only envelopes which expanded noticeably when placed in a chamber evacuated by a water aspirator were used in the trials. The treatment of the tubes and sealing in lead envelopes was done by hand at DREO.

The packets were stored for 3, 6 or 9 months at 65°C and ambient humidity. Evaluation was performed by adding a drop of 2M sodium hydroxide to the silica gel, as in the test for mustard. Any appearance of a light blue colour constituted a false positive test for mustard and indicated a useless tube.

RESULTS AND DISCUSSIONN-Ethyl-N-(2-hydroxyethyl)anilineIsothermal Storage

Analysis of the aged samples proceeded smoothly, the observed retention times being identical to those of authentic samples of methanol and amine. Table I lists the composition of each of two ampoules stored in each way, with duplicate determinations being reported. For methanol and the amine the figures represent peak area multiplied by the detector response factor and normalized such that these two figures total 100%; for the impurity (of unknown response factor, of course) the reported figure is its peak area divided by the sum of the three uncorrected peak areas. A good measure of the amount of decomposition is the increase of the area of the impurity peak from beginning to end of the trial, each peak area having been normalized by the appropriate percentage concentration of amine, as listed in the Table.

The trace of methanol in the neat amine samples was presumably due to not cleaning the ampoule-sealing apparatus before switching to neat amine. The water content was less than 0.05% for all of the samples analysed.

The samples stored at 65°C exhibit, at worst, an increase of impurity (normalized by the amine concentration) of 1%. Since six months storage at 65°C is usually accepted as equivalent to five years at room temperature we conclude that ampoules sealed in this manner are completely

acceptable for storage of this amine or its methanol solution.

The motivation for working with the neat amine was to provide an alternative in case the amine-methanol solution failed the storage trials. Clearly this has proved unnecessary. In fact, the programmed temperature trials indicate that the decomposition reaction proceeds by the same mechanism regardless of the presence of methanol.

Sunlight promotes the decomposition reaction by a factor of three to four compared to the reaction at room temperature in the dark. Thus sunlight should be excluded.

The abrupt variations in methanol concentration are probably best attributed to loss of methanol through microfissures in the ampoule. At 65°C the vapour pressure of methanol is 760 Torr, so the ampoules withstand one atmosphere pressure difference. Occasional loss of methanol is not surprising. No loss of amine was expected since its vapour pressure is only about 0.1 Torr at 65°C.

Identification of the impurity was not considered warranted at this time, even though it would have made this study more complete by permitting determination of its GC response factor. It is conceivable that high concentrations might interfere more than proportionally with phosgene detection. This was thought to be unlikely and, since the impurity concentration is so small, can safely be ignored.

The analyses reported here and earlier (7) were all performed with the same Tenax-GC chromatographic column, representing a year of intermittent use. In the current work peak broadening, especially for the amine, baseline drift, and increased retention times were all noticed. These were considered to be the result of column deterioration: further work would require a new column.

Programmed Temperature Storage

Accelerated storage trials in which the temperature is increased linearly with time can provide more precise and useful information than simple isothermal trials (9). Such a trial was undertaken with this amine to obtain definite information on its storage characteristics.

The problem with isothermal storage lies in the extrapolation of the results. In this trial it was found that the amine decomposed no more than 1% during six months at 65°C. The real question, however, was to find the decomposition in five years at room temperature. Isothermal storage provides insufficient information to answer this definitely. The kinetics of many chemical reactions are described by

$$-dc_1/dt = kc_1^n c_2^m c_3^p \quad (1)$$

where c_i is the concentration of compound i

n, m, p, \dots are small numbers

k is the rate constant, given by

$$k = A \exp(-E/RT) \quad (2)$$

where A is the pre-exponential factor

E is the activation energy

R is the gas constant

T is the temperature

Most decomposition reactions are first order with respect to the compound undergoing decomposition, and thus

$$-dc/dt = kc = Ac \exp(-E/RT) \quad (3)$$

where c is the concentration of the decomposing compound. Clearly a single measurement of the progress of reaction over a period of time is insufficient to determine the two unknowns, A and E , of Eq. 3. For example, 10% decomposition in six months at 65°C is consistent with $E = 40$ kJ/mole, $A = 3.7 \times 10^5$ year⁻¹ or with $E = 10$ kJ/mole, $A = 7.4$ year⁻¹. The former pair predicts 11% decomposition in five years at room temperature, probably acceptable, the latter 57%, probably unacceptable. More dramatic examples are possible.

Storage under conditions of linearly increasing temperature permit the determination of E and A . The theory has been described by Morrison (9). The results of GSC analysis of our samples, stored in temperatures increased by one degree per week, are listed in Table II. The impurity concentration, ignoring response factor, as a fraction of the amine concentration, was used to calculate $f(c) = \ln(c_0/c)$ where c_0 is the initial concentration of the amine (Eq. 5 of Ref. 9). This was done for each pair of ampoules, and the results plotted against the appropriate temperature (allowing for the step function increases) in Figure 1. The straight lines in these graphs are the least-squares linear fits; E and A were determined from their slopes and intercepts following Morrison (9). The resulting values, and the percentage impurity that would be predicted after storage, are listed in Table III. The error analysis will not be reproduced here, but the experimental errors in k and thus in the amount of decomposition are close to an order of magnitude (arising from values with moderate errors appearing in exponentials).

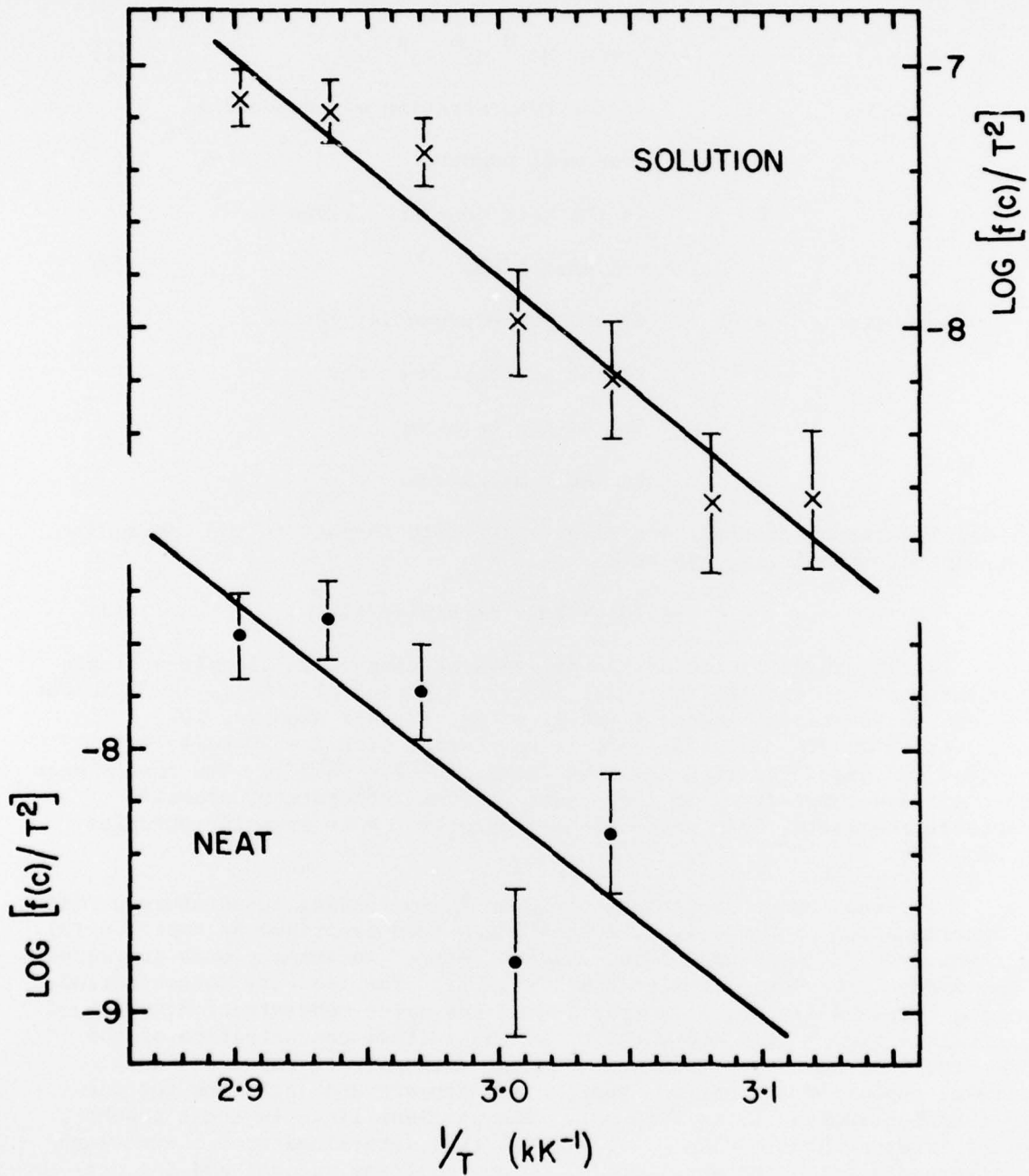


Figure 1. Graphs used to determine E and A from programmed temperature trials

The consistency of these results is very good, considering the slow pace of this reaction and the corresponding large experimental errors. The linear correlation coefficients for the plots are 0.97 and 0.80 for the solution and the neat amine, so the fit to a straight line is good. Within experimental error the activation energies are equal. The disagreement in the value of the pre-exponential factor, A, is probably not significant. In both cases the calculated amount of decomposition during six months at 65°C agreed, within experimental error, with the results of the isothermal experiments discussed above.

Two conclusions follow from these results. Clearly the amount of decomposition during five years at room temperature is very small, thus the ampoules are entirely adequate for storage of this compound. Secondly, it appears, since the activation energies and pre-exponential factors are the same for the neat amine and its methanol solution, that the decomposition reaction follows the same mechanism in the two cases.

"Blue-Band" Tubes

The storage problems with the "blue-band" tubes are not associated with the decomposition of the compounds on the silica gel, which are in crystalline form, but rather with the decomposition of trace substances on the gel which can produce false positive responses. The most demanding application of these tubes is the detection of mustard gas, which gives a faint blue colour. Thus in this trial the tubes were examined by adding a drop of base to the gel, as is done for mustard detection, and counting the number of tubes exhibiting blue traces.

In no case, after 3,6, or 9 months storage, were the blue colours intense. However tubes which still possessed their blue band exhibited sufficient blue tinge to block mustard detection near the required sensitivity levels in three to five of the tubes in each packet. In no case were such false positives found if the blue bands had been removed from all the tubes in the packet.

It was concluded that, not surprisingly, pigment from the blue band was diffusing into the atmosphere in the envelope and eventually onto the silica gel. This problem could easily be avoided by removing the bands before packaging.

Our recommendation, therefore, is that these blue bands be removed (In anticipation of this the descriptor "blue-band" has been in quotation marks in this note). An additional advantage of removing the bands is that the tubes will be less likely to stick in their holder. The bands are not required in the C2 kit, unlike previous kits, since no other tubes are used for detection.

CONCLUSIONS

When sealed in glass ampoules containing nitrogen, with normal precautions to exclude water, N-ethyl-N-(2-hydroxyethyl)aniline, either neat or in methanol solution, should decompose by considerably less than 1% during five years storage at room temperature.

Provided the painted blue band is not present, "blue-band" tubes sealed in lead envelopes with soldered seams should not give noticeable false positive tests after five years storage at room temperature.

Accelerated storage trials during which the temperature increases linearly with time produce more accurate and more useful information about storage lifetimes than do isothermal trials.

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TABLE II
 Percentage* Composition of Contents of Ampoules After Programmed-Temperature Storage Trials

| Sample Number | Storage Time (weeks) | | | | | | | | | | | | | |
|--------------------|---------------------------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| | 4 | | 8 | | 12 | | 16 | | 20 | | 24 | | 28 | |
| Component | 1 | 2 | 1 | 2 | 1 | 2 | 1 | 2 | 1 | 2 | 1 | 2 | 1 | 2 |
| | Solution samples [†] | | | | | | | | | | | | | |
| Methanol | 21.86 | 21.51 | 24.42 | 25.69 | 25.59 | 25.23 | 27.27 | 25.13 | 23.70 | 23.71 | 25.25 | 22.78 | 22.66 | 23.22 |
| Impurity | 0.30 | 0.32 | 0.29 | 0.29 | 0.31 | 0.31 | 0.30 | 0.32 | 0.59 | 0.46 | 0.54 | 0.59 | 0.61 | 0.53 |
| Amine [‡] | 78.14 | 78.49 | 75.58 | 74.31 | 74.41 | 74.77 | 72.73 | 74.87 | 76.30 | 76.29 | 74.75 | 77.28 | 77.34 | 76.78 |
| Decomposition | 0.01% | | 0.01% | | 0.02% | | 0.04% | | 0.26% | | 0.33% | | 0.38% | |
| | Neat amine samples [‡] | | | | | | | | | | | | | |
| Methanol | 2.15 | 1.14 | 0.97 | 2.98 | 1.05 | 1.01 | 2.30 | 2.28 | 1.06 | 2.52 | 1.67 | 1.02 | 1.32 | 1.32 |
| Impurity | 0.43 | 0.41 | 0.41 | 0.40 | 0.57 | 0.46 | 0.46 | 0.45 | 0.94 | 0.77 | 1.50 | 1.05 | 1.08 | 1.18 |
| Amine [‡] | 97.85 | 98.86 | 99.03 | 97.02 | 98.95 | 98.99 | 97.70 | 97.72 | 98.94 | 97.48 | 98.33 | 98.98 | 98.68 | 98.68 |
| Decomposition | 0 | | 0 | | 0.11% | | 0.04% | | 0.46% | | 0.85% | | 0.73% | |

* See text

† Solution of N-ethyl-N-(2-hydroxyethyl)aniline in methanol

‡ N-Ethyl-N-(2-hydroxyethyl)aniline

TABLE III

Results of Programmed Temperature Trials

| | Activation Energy, E | | Pre-Exponential Factor, A | |
|----------------------------|----------------------|-------------|---------------------------|--|
| | (kJ/mole) | (kcal/mole) | (h ⁻¹) ± 100% | |
| Amine in Methanol Solution | 159 ± 5 | 38 ± 1 | 4 × 10 ¹⁹ | |
| Neat Amine | 152 ± 8 | 36 ± 2 | 2 × 10 ¹⁸ | |

| | k [*] at 65 °C (h ⁻¹) | Decomposition [*] in 28 weeks | | k [*] at 20 °C (h ⁻¹) | Decomposition [*] in 5 years (Predicted) |
|----------------------------|--|---|-------|--|---|
| | | Predicted | Found | | |
| Amine in Methanol Solution | 5 × 10 ⁻⁶ | 3% | 0.5% | 2 × 10 ⁻⁹ | 0.01% |
| Neat Amine | 3 × 10 ⁻⁶ | 1% | 0.4% | 6 × 10 ⁻¹⁰ | 0.003% |

* See text for error discussion

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KEY WORDS

**Storage Trials
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Detector Kit, Chemical Agent, C2
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