Appendix 1

VX Chemical Considerations

2 February 2016

Background

- VX is a complex, 17-component engineered formulation that is designed around creating specific chemical interactions between its various components
- It is specified to be utilized in concentrations varying from 5-20%
- VX has enjoyed only limited commercial utility and is currently being discontinued
- The major factor limiting the utility of VX is its lack of stability, especially outside of its 1 year recommended shelf life
  - Chemical degradation contrary to study conclusion (p.33)
    - Known, demonstrable differences by FTIR between fresh and 11 month samples of VX
      - FTIR spectra from 6 month samples from the 2-year studies were requested and denied
    - Demonstrated depletion of key formulation components versus time by GC/MS and HPLC
    - Sample settling notable by SG
- Significant differences were noted in the as-measured components listed in Table 1 p.34 of the Peer Review Draft and the actual components contained in a fresh sample of VX

VX Study - Analytical Concerns

- The analysis of the two VX samples as reported in Table 1 p.34 identified some, but not all, of the 17 manufacturing materials contained in a fresh sample of VX
  - The analysis as reported identified 13 of the 17 compounds and a complex hexane extract
    - 8/13 of the materials identified were identical/nearly identical to those in the as-manufactured product
    - The content of the hexane extract was not identified, but likely contained 4 key components of the manufactured material
    - The analysis did not detect 4 other critical components of the manufactured material
  - In those 8 instances where the materials were identified qualitatively, the quantities shown varied substantially in some cases from the manufactured composition of the VX
- The general conclusion is that the two lots of material that were tested were not chemically equivalent to the VX as produced and marketed by Master Chemical Corporation
  - The study is invalid as to VX and any generalized conclusions about it or other soluble oil metal working fluids

Appendices

Appendix 1. Aging Studies

Sample Description	Sample ID	Comments
Top of freshly made lab control	WB3201-126A-T	Made by BJM on 1/11/2016
Bottom of freshly made lab control	WB3201-126A-B	Made by BJM on 1/11/2016
Top of oldest QC retain sample	Lot 022515H-T	QC retain from 02/25/2015
Bottom of oldest QC retain sample	Lot 022515H-B	QC retain from 02/25/2015
Top of oldest undisturbed drum	Lot 071015H – UT	Manufactured 07/10/2015
Bottom of oldest undisturbed drum	Lot 071015H – UB	Manufactured 07/10/2015
Top of oldest undisturbed drum mixed 5 minutes	Lot 071015H – M5T	Manufactured 07/10/2015
Bottom of oldest undisturbed drum mixed 5 minutes	Lot 071015H – M5B	Manufactured 07/10/2015
Top of oldest undisturbed drum mixed 1 hour	Lot 071015H – M1HT	Manufactured 07/10/2015
Bottom of oldest undisturbed drum mixed 1 hour	Lot 071015H – M1HB	Manufactured 07/10/2015

FTIR Aging Study Key Result

"Eight of the samples gave similar FTIRs. Two of the samples (**Lot 022515H-T** and **Lot 022515H-B**) were slightly different from the other eight. The formation of a new peak, although slight, can be seen at 1710 cm^{-1}. The appearance of this peak at 1710 cm^{-1} is not unusual considering **Lot 022515H-T** and **Lot 022515H-B** are the oldest samples in the group. Peaks in that area of the FTIR spectrum refer to carbonyl stretches, most likely of carboxylic acids or esters. The fact that ester or acid is changing over time is not that unusual and is probably occurring in all ten of the samples. However in the two samples, the change has occurred to a greater extent, allowing the change to be seen in the FTIR."*

GC/MS Aging Study Key Result

"In these experiments, a new batch of Trim VX (WB3201-126A) was compared to an older batch (Lot 022515H). . When the experiment was conducted by GC/MS, it was established that the 4-chloro-3-methyl-phenol depleted by 2 % ."*

*Results from MCC Technical Report JHM January 19, 2016 "Test Protocol to Determine Stability of TRIM® VX Concentrate over Time"

Appendix 1b

January 19, 2016

Steve,

Please note the Specific Gravity from the top and bottom of the oldest and newest plant manufactured Quality Control retains. We ran the bottom sample twice for Lot # 081915N just to double check because the value was unusually high. In my experience a variation of greater than +/- 0.01 units is a concern.

Best regards,

Brian

VX 022515H
Top = 1.001
Bottom = 0.998

VX 081915N
Top = 1.003
Bottom run #1 = 1.033
Bottom run # 2 = 1.031

Appendix 2. Compositional Analysis

Reverse Engineered Chemistry Identified	Lot 101607N	Lot 011509N	Manufacturing Component?	% Delta v Manufactured VX: Lot 101607N	% Delta v Manufactured VX: Lot 011509N
Water	7.1	6.8	Yes	39	33
Triethanolamine	3.7	3.2	Yes	19	2
4-Chloro-3-methyl-phenol	3.59	2.49	Yes	88	31
Diethylene glycol	0.87	1.07	No****	NA	NA
Diethylene glycol monobutyl ether	1.02	1.11	No ****	NA	NA
Methyl palmitate	1.18	1.2	Yes*	1	4
Methyl oleate	5.65	5.81			
Methyl stearate	0.89	0.93			
Myristic acid	0.49	0.23	No ****	NA	NA
Oleic acid	3.18	1.23	Yes	-20	-69
Palmitic acid	1.01	0.31	No ****	NA	NA
Propylene glycol	0.2	0.2	No ****	NA	NA
α-Terpineol	0.6	0.5	Yes**	0	-17
Hexane extractable material	80.2	85	Yes***	17.6	24.6

* Manufacturing raw material varies from lot to lot in terms of specific amounts of esters

** α-Terpineol is the key component of the manufacturing raw material

***4 specific manufacturing materials not identified

****The 5 materials identified by NTP that were not specifically added by Master Chemical Corporation to the VX formulation may have been present in some unknown amount as secondary/trace products contained in the intended raw material