



Solvent/Water Partitioning and Extraction of Dimethyl Methylphosphonate

Importance of Hydrogen-Bonding

Daniel C. Leggett

June 1990

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Special Report 90-21



**U.S. Army Corps
of Engineers**
Cold Regions Research &
Engineering Laboratory

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Prepared for
OFFICE OF THE CHIEF OF ENGINEERS

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PREFACE

This report was prepared by Daniel C. Leggett, Research Chemist, of the Geochemical Sciences Branch, Research Division, U.S. Army Cold Regions Research and Engineering Laboratory. Funding for this research was provided by DA Project 4A161102AT24, *Research in Snow, Ice and Frozen Ground*, Task SS, Work Units 031, *Chemical Persistence at Low Temperatures*, and 020, *Chemical Species Transport Phenomena in Frozen Ground*.

The author thanks Alan Hewitt and Dr. Patrick Black of CRREL for their technical review of this report.

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DANIEL C. LEGGETT

INTRODUCTION

In addition to its being a commercial flame retardant and viscosity suppressant (Howard et al. 1986) dimethyl methylphosphonate (DMMP) is widely used as a nontoxic simulant for G-agents by the U.S. Army and its contractors (Bennett et al. 1984). DMMP is completely miscible with water at room temperature and has a projected hydrolysis half-life under neutral conditions of 88 years (Mabey and Mill 1978) but is susceptible to both acid- and base-catalyzed hydrolysis. Its octanol-water partition coefficient (P) is 0.074 (Leggett 1987), which suggests that its movement in groundwater aquifers would be relatively unimpeded (Wilson et al. 1981, Lyman 1982). Commercial and Army use of this compound, coupled with its chemical stability and propensity to migrate with groundwater flow, suggest that it is a probable groundwater contaminant. Thus it is likely that a method sensitive enough for groundwater monitoring of DMMP and similar compounds will be needed.

Existing methods rely on direct aqueous injection (Leggett 1987, 1988) gas chromatography (GC) or solvent extraction with chlorinated solvents* followed by GC analysis. Direct aqueous injection GC is reliable but relatively insensitive, while the solvent extraction methods used to date suffer from poor recoveries.* Recent work showed that several water-miscible polar solvents can be used for solvent extraction of polar solutes by salting out (Hertz 1989, Leggett et al. 1990). Our guiding assumption was that polar solvents would be better at extracting polar compounds. This appears to be generally true; however, for polynitro-organics the most critical property of the solvent was found to be its basicity. Electron-pair donor solvents (Lewis bases) stronger than water were required to extract the electron-deficient, Lewis acid nitro-organics (Leggett et al. in prep.). Analogous reasoning suggested that since DMMP is a Lewis base it would be extracted from water by Lewis acids. For technical reasons I was unable to test this idea, but found that hydrogen-donor (Bronsted acid) solvents worked well. That observation forms the

basis for this report. I have been able to examine the partitioning of DMMP between water and various H-donor, H-acceptor, and neutral solvents, with the help of salting to separate several miscible solvents, and relate this to H-bond formation between DMMP and the solvent and preferential solvation of the H-bonded complex.

Another reason for selecting DMMP for this work was that it has the lowest octanol-water partition coefficient (P) of compounds in this series (Leggett 1987, Krikorian et al. 1987) and therefore should be the most difficult to extract with organic solvents. The positive relationships (Linear Free Energy Relationships, LFERs) among partition coefficients in various organic solvents (Leo et al. 1971) virtually assure that any solvent found suitable for extracting DMMP from water will be even better for extracting higher molecular weight phosphonates. For example, diisopropyl methylphosphonate (DIMP), which is also of environmental concern (Howard et al. 1986), and the G and V agents (GB, GD, VX, etc.) all have higher log P values than DMMP (Leggett 1987). Lastly, it is hoped that this work contributes to an understanding of the chemistry of phosphonates, which are typified by DMMP.

EXPERIMENTAL

Solvents used in partitioning were of various grades, obtained from several suppliers and used without further purification. DMMP was obtained from Aldrich Chemical Co. A stock solution of DMMP was prepared by adding 100 μ L of the liquid to 200 mL of house distilled water and mixing thoroughly. Five mL of this solution was pipetted into a 20-mL liquid scintillation vial followed by 5 mL of the test solvent. In salting experiments an excess of NaCl (Baker) was added after the solvent. The vials were sealed with polyethylene-lined caps and shaken vigorously for several minutes. The phases were allowed to separate (from minutes to hours depending on the rate of separation). In most cases both phases were analyzed by direct injection of 2- μ L aliquots into the gas chromatograph. In a few instances, one phase was determined by the difference relative to the mean of a number of injections of the

*Personal communication with M. Stutz, U.S. Army Toxic and Hazardous Materials Agency, 1989.

stock DMMP solution. The coefficient of variation of these standard injections was less than 5%. A preliminary check of the sums of the concentrations of DMMP in the two phases was performed to ensure freedom from interferences. Although the concentration values are not strictly additive because of phase volume changes, these variations were less than $\pm 20\%$. Positive deviations greater than this were considered to result from interferences in the chromatography and the data were discarded. Solvents for which this occurred were nitrobenzene, benzyl alcohol, and toluene. There were no significant negative deviations in the data.

Gas chromatography was performed on a Perkin-Elmer Model Sigma 2 GC equipped with a flame ionization detector. The glass analytical column was 6-ft \times 1/8-in.-o.d. and filled with 10% SP-1000 (Supelco) on 100/120 mesh (Supelcoport) maintained at 115°C. The carrier was zero-grade N₂ with an in-line oxygen stripper (J & W) at a flow rate of 20 mL/minute. Injector and detector were maintained at 200°C and 250°C, respectively. Data were taken with a Hewlett-Packard 3390 digital integrator. The retention time of DMMP under these conditions was 8.0 minutes.

FORMAT

The terms "partitioning" and "extraction" are used interchangeably in this report, depending on the emphasis. The data are expressed as partition coefficients because these are the more fundamental measures related to the thermodynamics of phase equilibria. A theoretical extraction efficiency can always be calculated, once the partition coefficient is known, from the equations:

$$P_{sw} = \frac{C_s}{C_w} \quad (1)$$

$$\% \text{ extracted} = \frac{C_s V_s \cdot 100}{C_s V_s + C_w V_w} \quad (2)$$

where P_{sw} is the partition coefficient, C_s and C_w are concentrations of the solvent and water, respectively, and V_s and V_w are the final volumes of solvent and water, respectively.

Combining equations:

$$\% \text{ extracted} = \frac{V_s \cdot 100}{V_s + \frac{V_w}{P_{sw}}} \text{ or } \frac{P_{sw} V_s \cdot 100}{P_{sw} V_s + V_w} \quad (3)$$

Note that the percentage extracted is uniquely determined by the volumes of water and extracting solvent, which can be adjusted (once P_{sw} is known) to suit the analyst and his analytical requirements.

RESULTS

Table 1 is a summary of the experimental data. Solvents are listed in the order of increasing partition coefficient ($\ln K$) of DMMP between it and water. The partition coefficients for DMMP between each solvent and NaCl-saturated water are also given. The log P values for water-miscible solvents without salt shown in parentheses were calculated on the basis of the average salting-out coefficient of the other solvents for which direct measurements were made, which was 0.86 ± 0.13 log units or a factor of 7; i.e., on average the solvents extracted seven times as much DMMP from salt-saturated water as from distilled water. Also shown in Table 1 is the standard free energy of transfer of DMMP from water to each organic solvent, which was calculated from the relation:

$$\Delta G^\circ = -RT \ln K_{eq} \quad (4)$$

where ΔG° = standard free energy

R = the gas constant

T = absolute temperature

K_{eq} = the equilibrium constant in mole fraction units.

The ordinary volume-based partition coefficients (P) in Table 1 were converted to mole fraction form (K). This was done by multiplying by the ratio of molar volumes of solvent to water as calculated from their molecular weights and liquid densities. These data and the molar volumes are shown in Table 2.

DISCUSSION

Most of the tested solvents failed to extract DMMP from water efficiently, as indicated by their negative log P values. In the absence of salt only three of the tested solvents had log P values greater than 0: chloroform, trifluoroethanol (TFE), and hexafluoroisopropanol (HFIP). P for the latter was out of the range of measurement in this experiment because the residual aqueous phase concentration was undetectable. In the presence of saturated salt, however, many other solvents had positive log P s. This fact demonstrates the utility of adding salt for the practical purpose of extracting DMMP from water. In the absence of salt, chloroform was only a fair extractant, $P = 1.5$. Addition of salt increased this to 9.3. The best solvents, TFE and HFIP, are miscible in the absence of salt, so for efficient extraction of DMMP one has no choice but to use salt.

Inspection of Table 1 also shows that the most

Table 1. Partitioning of DMMP between water and various solvents.

<i>Solvent</i>	$\log P^*$	$\log P_{sat}^*$	$\ln K_{eq}^\dagger$	ΔG°^{**}
Hexane	(-3.32)††	-2.46	(-5.66)	(3300)
Carbon disulfide	-2.58	-1.48	-4.73	2760
Carbon tetrachloride	-1.94	-1.00	-2.78	1620
Benzene	-1.61	-0.67	-2.11	1230
1,1,1-Trichloroethane	(-1.56)	-0.70	(-1.89)	(1100)
Diethylether	-1.49	-0.73	-1.66	970
Trichloroethylene	-1.27	-0.33	-1.31	760
Tetrahydrofuran	(-1.09)	-0.23	(-1.00)	(590)
Acetone	(-0.84)	0.02	(-0.53)	(310)
n-Octanol	-1.13	(-0.27)	-0.42	240
Benzonitrile	-0.81	0.01	-0.12	70
Ethyl acetate	-0.78	0.22	-0.10	60
Methylethylketone	-0.73	-0.08	-0.08	50
Water	0.00	(0.86)	0.00	0
Acetonitrile	(-0.44)	0.42	(0.05)	(-30)
Nitroethane	-0.50	0.17	0.21	-130
Isopropanol	(-0.46)	0.40	(0.39)	(-220)
Nitromethane	-0.25	0.49	0.52	-310
Propylene carbonate	-0.44	0.065	0.53	-310
Benzyl nitrile	-0.52	0.09	0.62	-380
n-Propanol	(-0.31)	0.55	(0.71)	(-410)
Propanol	-0.26	0.10	0.79	-460
Bromoforn	-0.34	0.52	0.80	-470
Methylene chloride	-0.18	0.66	0.84	-490
1,1,2-Trichloroethane	-0.31	0.47	0.91	-530
Isobutanol	-0.13	0.51	1.34	-780
Chloroform	0.17	0.97	1.88	-1100
2,2,2-Trifluoroethanol	(1.32)	2.18	(4.44)	(-2590)
Hexafluoroisopropanol	(>1.56)	>2.42	(>5.36)	(<-3130)

**P* in dimensionless *V/V* units as tabulated in Leo et al. (1971).

†*K* in dimensionless mole fraction units.

** ΔG° in cal/mole.

†† Parenthetical values are theoretical computations, not experimental.

effective extraction solvents are alcohols and halogenated solvents, which are known H-bonders (Pimentel and McClellan 1960). One may take special note of the remarkable difference in $\log P$ of the two trichloroethane isomers and the smaller but significant difference between benzo- and benzyl nitrile. Both these examples show clearly the effect of electronegative substituents on the activation of H in the methane acids. The least effective extraction solvents are neutral, non-polar compounds. Intermediate are polar electron-pair donors, which are actually amphoteric, i.e., can either donate or accept electrons. These data are consistent with the reported differential heats of mixing of DMMP between several solvents and water (Chang et al. 1987) that I have plotted in Figure 1. Unfortunately, there were too few data for a definitive comparison, but the trend is clear.

The partition coefficients are also comparable to measured complex formation constants reported for

sarin (GB) with substituted phenols and trimethyl phosphate with trifluoroethanol (Higuchi et al. 1969, Ruosteso et al. 1987). In fact Higuchi et al.'s data for sarin were obtained by liquid partition of the H-donor complexes into cyclohexane. This suggests that it may be possible to extract DMMP into a nonpolar solvent in the presence of strong H-donors such as HFIP. The similar ΔG° values for complex formation of the above compounds (Higuchi et al. 1969, Ruosteso et al. 1987) and solvent extraction of DMMP strongly suggest that the driving mechanism for both is the formation of H-bond, charge-transfer complexes. This explanation is further supported by the spectral and enthalpy of mixing data (Chang et al. 1987, Barlow et al. 1987).

Butanol seems to have an anomalously high partition coefficient relative to its reported heat of mixing with DMMP, which would seem to imply that the partitioning of DMMP into butanol is due more to differential solvation of the DMMP-H₂O complex than to H-bond-

Table 2. Molecular weights, densities, and molar volumes of solvents used in partitioning experiments.

<i>Solvent</i>	<i>MW*</i>	<i>d</i> †	<i>V_m**</i>
Acetone	58	791	73.5
Acetonitrile	41	783	52.3
Benzene	78	879	88.5
Benzonitrile	103	1000	103.0
Benzyl nitrile	117	1020	115.0
Bromoform	253	2890	88.0
Carbon disulfide	76	1260	60.2
Carbon tetrachloride	154	1590	97.1
Chloroform	119	1490	80.0
Diethyl ether	74	708	104.0
Ethyl acetate	88	901	98.0
Hexane	86	659	131.0
Hexafluoroisopropanol	168	1600	105.0
Isobutanol	74	802	92.6
Isopropanol	60	785	76.3
Methylene chloride	85	1340	63.3
Methylethyl Ketone	72	805	89.3
n-Octanol	130	829	157.0
n-Propanol	60	804	74.6
Nitroethane	75	1050	71.4
Nitromethane	61	1130	54.1
Propanol	58	807	71.9
Propylene carbonate	102	1190	85.4
Tetrahydrofuran	72	888	81.3
1,1,1-Trichloroethane	133	1350	99.0
1,1,2-Trichloroethane	133	1440	92.6
Trichloroethylene	131	1470	89.3
2,2,2-Trifluoroethanol	100	1370	73.0
Water	18	1000	18.0

*Molar weight in grams.

†Density at 20° or 25°C in g/L.

**Molar volume in mL/mole.

ing. This is probably true of the other unsubstituted alcohols as well, and is consistent with these alcohols being slightly weaker proton donors than water (Albert and Serjeant 1962, Taft et al. 1985). It is not evident why chloroform and methylene chloride interact more strongly with DMMP than the alcohols, since they are supposedly even weaker acids, while nitromethane and nitroethane, which are much stronger acids in water, fail to extract DMMP as efficiently as either the halogen acids or the alcohols. Table 3 summarizes the pK_a values of solvents that are acids relative to water. Obviously there is no correlation with $\ln K_{eq}$, considering this criterion alone. Similar conclusions have been drawn with respect to pK_a values of organic bases and formation constants of complexes with reference acids (Gurka and Taft 1969). The correlation seems to be better when comparing only compounds within a single class (Higuchi et al. 1969, Ruosteso et al. 1987). Steric requirements for bond formation and self-association of the solvent conceivably interfere with complex forma-

tion. One would expect nitromethane, nitroethane and other amphoteric solvents to self-associate, which would lower their interaction energy with trace solutes.

An estimate of the hydrophobic contribution to water \rightarrow solvent transfer can be obtained from the intercept of the line in Figure 1; i.e., when the differential heat of mixing is 0, $\ln K_{eq} = 1.4$. Since $\Delta G^\circ = -RT \ln K_{eq} = \Delta H^\circ$

Table 3. Acid dissociation constants and solvent/water partition coefficients of DMMP.

<i>Solvent</i>	<i>pK_a</i>	<i>ln K_{eq}</i>
Nitroethane	8.4	.21
Hexafluoroisopropanol	9.3	>5.36
Nitromethane	10.2	0.52
Trifluoroethanol	12.4	4.44
Water	15.7	0.0

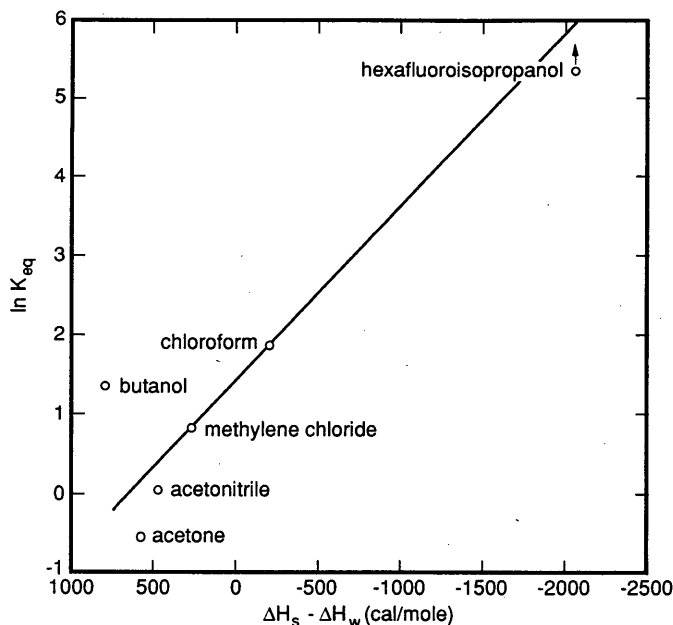


Figure 1. DMMP partition between water and organic solvents vs differential heats of mixing with these solvents.

$-T\Delta S^\circ$, when the enthalpy change is 0, $T\Delta S^\circ = RT \ln K_{eq} = 820$ cal/mole, the standard entropy contribution to the free energy at the experimental temperature of 294 K, or about 3 cal/mole-K. In contrast, typical entropies for hydrocarbon (hydrophobic) transfers from water to organic solvents are 20 cal/mole-K (Leo et al. 1971, Tanford 1980), a further indication that stronger interactions are involved in the present work.

In summary, the extraction of DMMP from water with various solvents was related to the propensity of donor solvents to form hydrogen bonds with it, although there was no direct correlation with pK_a of the solvent. The successful extraction of DMMP from water seems to depend on formation of an H-bonded complex which is then extracted by the solvent. The fluorinated alcohols, trifluoroethanol, and hexafluoroisopropanol, proved to be superior to other H-bond donors tested, regardless of their acid strength in water. Since these two solvents are miscible with water, it was necessary to add salt to obtain separation. Salting has the added advantage, moreover, of increasing the apparent partition coefficient by a factor of 7 relative to unsalted systems for all solvents. The obvious practical conclusion of this work is that trace concentrations of DMMP and similar compounds in water can be preconcentrated (> 100-fold) by salting-out solvent extraction with

HFIP or TFE. I would even go so far as to suggest that these solvents ought to have broad applicability for extraction of electron-pair donor compounds (bases) from water, which should be considered when developing analytical methods for such compounds as ketones, ethers, nitriles and esters.

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REPORT DOCUMENTATION PAGE

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1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE June 1990		3. REPORT TYPE AND DATES COVERED	
4. TITLE AND SUBTITLE Solvent/Water Partitioning and Extraction of Dimethyl Methylphosphonate: Importance of Hydrogen Bonding				5. FUNDING NUMBERS RE: 6.11.02A PR: 4A161102AT24 TA: SS WU: 020, 031	
6. AUTHORS Daniel C. Leggett					
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) U.S. Army Cold Regions Research and Engineering Laboratory 72 Lyme Road Hanover, New Hampshire 03755-1290				8. PERFORMING ORGANIZATION REPORT NUMBER Special Report 90-21	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Office of the Chief of Engineers Washington, D.C. 20314-1000				10. SPONSORING/MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES					
12a. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution is unlimited. Available from NTIS, Springfield, Virginia 22161				12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) Partition coefficients for dimethyl methylphosphonate (DMMP) between water or salt-saturated water and 28 organic solvents were measured. On the average the solvents extracted 7 times as much DMMP from sodium chloride-saturated water as from distilled water. The highest partition coefficients were with H-donor solvents, especially the fluorinated alcohols, trifluoroethanol and hexafluoroisopropanol. Partition coefficients for these solvents and salt-saturated water were 150 and >260, respectively. These results indicate that DMMP can be extractively preconcentrated >100-fold from water with an appropriate choice of solvent and addition of excess sodium chloride. Since DMMP is the lowest homolog of the phosphonate series, higher members are expected to have higher partition coefficients because of greater hydrophobic contributions. The principal mechanism driving extraction appears to be formation of H-bond donor-acceptor complexes which are preferentially solvated by the solvent.					
14. SUBJECT TERMS Dimethyl methylphosphonate (DMMP) Partition coefficients Partitioning			Salting-out Solvent extraction		15. NUMBER OF PAGES 12
					16. PRICE CODE
17. SECURITY CLASSIFICATION OF REPORT UNCLASSIFIED	18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED	19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED	20. LIMITATION OF ABSTRACT UL		