

DECHLORINATION OF PCBs AND CAHs USING Na/NH_3 . APPLICATION TO SOIL REMEDIATION

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ABSTRACT

Remediating toxic compounds from soils prevents subsequent leaching into groundwater. Model halogenated aromatic, aliphatic and olefinic hydrocarbons and halogenated phenols were dehalogenated in seconds by solvated electrons generated from sodium in both anhydrous liquid ammonia and ammonia/water solutions. The minimum amount of sodium required to completely dehalogenate these model compounds was determined by increasing the Na/substrate ratio until halogen loss was complete. Minimum sodium consumptions were determined in both anhydrous liquid ammonia and with a (5, 20, 50-fold molar excess of water per mole of halide) present. While more Na was consumed in the presence of water, these dehalogenations were still efficient when a 50-fold water excess was present. Dehalogenation is faster than competing reactions with water. CCl_4 , CH_3CCl_3 and $\text{CCl}_2=\text{CCl}_2$ in the presence of a stoichiometric deficiency of sodium produced only CH_4 , CH_3CH_3 and ethylene and recovered CCl_4 , CH_3CCl_3 or $\text{CCl}_2=\text{CCl}_2$, respectively. No partially dechlorinated products were detected, indicating each dechlorination was diffusion controlled. Na consumption per chlorine removed (as NaCl) was lower than that of Li, K or Ca and this advantage increased in the presence of water. This work was extended to PCB- and CAH-contaminated soils (both wet and dry). PCBs were remediated with 99.9% and higher efficiencies by slurring soils in NH_3 followed by addition of sodium. Soils contaminated with CAHs were remediated from 4000 - 3000 ppm levels to ~1 ppm. The required sodium consumption per Cl removed was determined in both wet and dry soils. As the concentration of CH_3CCl_3 in soil decreased, larger amounts of sodium were required (per Cl removed) to further destroy the remaining CH_3CCl_3 . However, even in wet soils, the sodium consumption was not excessive. This method also destroys polynuclear

aromatic hydrocarbons (PAHs) and nitro compounds found in contaminated soils around munitions plants.

INTRODUCTION

PCBs and other chlorinated aromatic compounds are distributed in soils and sludges at over 400 sites in the USA. CAHs occur as serious contaminants at 358 major hazardous waste sites in the USA and they migrate vertically through soils to form DNAPLs on aquifer bottoms. Every state is represented in this problem. To give just one example, DOE's Hanford site has massive soil and groundwater contamination with carbon tetrachloride. A subsurface plume extends for over 70 sq. miles. Many contaminated sites exist in the Gulf Coast region (Texas through Florida) where the largest concentration of chemical manufacturing plants in North America is located together with many DOD sites. Thus a national need exists for both *ex-situ* and *in-situ* methods to destroy these pollutants in soils and sludges rapidly at ambient temperature. Successful remediation avoids later contamination of groundwater on a wider scale.

We have demonstrated that both Na/NH_3 and Ca/NH_3 solutions (solvated electrons) will dechlorinate PCBs and CAHs in seconds at ambient temperature even in the presence of excess water (Pittman and Mohammed 1996; Sun, He and Pittman 2000). PCB- and CAH-contaminated soils (as received clay, loam and sandy soils containing up to 25% water) were successfully decontaminated within 30 sec. at 25°C. PCB and CAH destruction efficiencies >99.9% were achieved. The major goal is to develop solvated electron chemistry (e.g. Na/NH_3) as a single, multifunctional, portable technology applicable to both on site *in-situ* and on site *ex-situ* destruction of PCBs, CAHs, pesticides, herbicides, chemical warfare agents

and munition/explosive residues. If only half of these classes of pollutants can be rapidly destroyed in solvated electron media, this single technology would have broad application.

Alkali metal-based reductive dehalogenation is a well known process. Sodium has been added to halogenated organic compounds without solvent but this heterogeneous reaction is slow and moisture sensitive (Knorre, Langer and Pohl 1980). Li, Na, K or Ca in liquid NH_3 generates solvated electrons which dehalogenate dissolved organic compounds at exceptionally fast rates (Jessup et. al. 1977; Mackenzie, Kopinke and Remmler 1986). Commodore Solution Technologies (Weinberg and Abel 1989, 1992) and our group (Pittman and Tabaei 1993) have shown that PCB-contaminated wet soils can be rapidly remediated with Na/NH_3 or Ca/NH_3 's, demonstrating these dechlorinations are much faster than reactions in water. The half-life ($t_{1/2}$) of solvated electrons in purified NH_3 is ca. 300h but in pure water the solvated electron has a $t_{1/2}$ of ca. 100 microsec. Dechlorination of PCB-contaminated wet soils is possible due to the relatively long solvated electron lifetime in 20% $\text{H}_2\text{O}/80\% \text{NH}_3$ ($t_{1/2} = \text{ca. } 100\text{sec.}$) coupled with fast dechlorination by $\text{Na}/\text{NH}_{3(\text{L})}$.

No systematic study has appeared of solvated electron dechlorinations in $\text{NH}_{3(\text{L})}$ with excess water present. PCBs, CAHs etc. intercalated deep in clay layers require diffusion of solvated electrons into soil or extraction of chlorinated organics into the bulk solution. This allows time for competitive reactions with water. Competitive reactions may accelerate in the presence of oxygen, transition metal ions, acidic soil sites etc. Therefore, the consumption of Na, required to achieve complete dechlorination of both chloroaromatic and chloroaliphatic compounds in the presence of excess water, was determined in this work and reported herein together with examples of soil remediation studies employing Na/NH_3 .

RESULTS AND DISCUSSION

Model Compound Studies

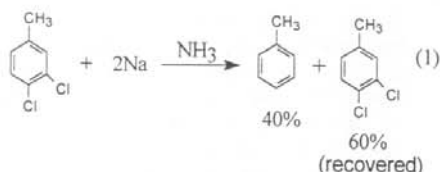
Twenty eight aliphatic and aromatic halogenated model compounds were completely dehalogenated in seconds by solvated electrons. These reductions

required only the time needed to dissolve the metal. The minimum amount of sodium required to completely dechlorinate both aromatic and aliphatic compounds was determined by the stepwise increase of the Na/substrate ratio in several reactions until complete chlorine loss ($>99.95\%$; to nondetectable levels) occurred. This ratio was obtained both in anhydrous liquid ammonia and in the presence of water (5, 20, 50-fold molar excess of water per mole of halide) as shown in Table 1 for monochloroaryl compounds (entries 1-3), di-, tri- and tetrachloroaryl compounds (entries 4-7), chlorinated phenols (entries 8-10) and chlorinated aliphatic compounds (entries 11-14).

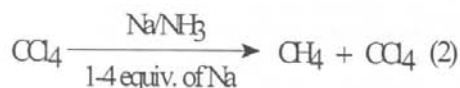
All dechlorinations required more than one equivalent of Na to complete in anhydrous $\text{NH}_{3(\text{L})}$. This might be due to (1) reaction of solvated electrons, with NH_3 to generate NH_2^- , catalyzed by traces of Fe^{+3} , (2) formation of aryl carbanions (after loss of chlorine) or (3) competing reactions with water (last 3 columns, Table 1). Aryl carbanion formation consumes two electrons per chloride lost. The most important finding is that adding substantial amounts of water did not greatly increase the amount of Na needed for complete dehalogenation. Increasing chlorine substitution leads to a decreased consumption of Na per chlorine lost, both with and without water present for both aromatic and aliphatic molecules. CCl_4 , CH_3CCl_3 and $\text{C}_6\text{H}_5\text{Cl}_5$ (lindane) required less sodium per chlorine lost than halogenated aromatic compounds, especially in the presence of excess water. In anhydrous ammonia, CCl_4 , CH_3CCl_3 and lindane required 1.15, 1.2 and 1.1 equiv. of Na per chlorine, respectively, for complete dechlorination.

Dechlorination With Deficient Amounts of Sodium

Partially dechlorinated intermediates were not observed when 1,2-dichlorobenzene, 3,4-dichlorotoluene, CCl_4 and CH_3CCl_3 were reduced in $\text{NH}_{3(\text{L})}$ with a Na deficiency (Table 2) either with or without H_2O present. For example, 3,4-dichlorotoluene (Equation 1) gave only toluene and recovered starting material when treated with Na/NH_3 at Na/substrate ratios of 1, 2 and 3 in the presence of 5 equivalents of water.



Dechlorinations of CCl_4 with 1, 2 and 3, 4 equivalents of Na in anhydrous NH_3 gave only C_2H_6 , recovered CCl_4 and traces of unknown products. The material balances were almost complete. No CHCl_3 , CH_2Cl_2 or CH_3Cl were detected by GC analysis (see equation 2).



Similarly, dechlorination of CH_3CCl_3 with 2 equivalents of Na produced ethane (62%) and CH_3CCl_3 (38%) was recovered unchanged. The material balances confirm that NH_3 or NH_2^- is not reacting with partially dechlorinated intermediates to give aminated products or elimination.

These results argue that the four dechlorinations in Table 2 are diffusion controlled. Apparently, solvated electrons react rapidly with partially dechlorinated species before these chlorine-containing intermediates can diffuse away to other regions of the solution and be replaced by more substrate. Reaction of solvated electrons with substrate goes to completion faster than substrate escape from the vicinity of the dissolving Na particle, despite rapid stirring. The supply of solvated electrons (Na dissolution) must also be fast enough to completely dechlorinate intermediates before they equilibrate with more substrate in the vicinity of the dissolving particle. Thus, dechlorination appears to be diffusion controlled. Hence, solvated electrons are consumed before mixing can equilibrate them with substrate. In these experiments particles of sodium were added to solutions of NH_3 and RCI .

Soil Remediation Studies

Laboratory and Commercial Scale Solvated Electron Technology Experiments on Soils

Contaminated soils (both prepared and from contaminated sites) were slurried in NH_3 at ambient temperature. After premixing, a weighed quantity of solid Na or Ca was dropped directly into the stirring slurry. The metal quickly dissolved. Conductivity and calorimetry showed the reactions were complete within a few seconds. Table 3 demonstrates the remediation of ~100 g soil samples by Ca/NH_3 (excess Ca). In clay, sandy or organic soils the destruction efficiencies were >99.9%. Similar studies were done with sodium.

These treatments are being scaled up and several process variations have been developed depending on the nature of the material being remediated. Soils from each particular remediation site have repeatedly shown that Na was more effective than Ca. Na achieves the highest cost effectiveness. Front-end treatment to remove water or extract contaminants can be used. A solvated electron treatment module is the centerpiece of each process. Back end processes to recycle NH_3 , adjust pH and concentrate or fix the reaction products have been developed. A commercial 1200 liter unit is now available (Commodore Solution Technologies) to treat PCBs, CAHs, etc. in oil, liquid pesticides or contaminants which have been extracted from soil, sludges, etc. Most commercial soil decontaminations operate via extraction by NH_3 first, followed by decontamination with Na.

CB-Contaminated Soils

A wide variety of soils have now been treated. Soil characteristics which can impact the solvated electron chemistry include loam, sand, silt, clay, humic material, pH, cation exchange capacity, particle size, water, and iron content. Processes have been engineered to accommodate this wide range of variables. Some soils can be treated as-received. Others require pre- or post-processing (e.g. water removal, size reduction, washing, and pH adjustment) to effectively remediate them.

Processes can be modified to deliver targeted remediation levels. Many different soil contaminants have been treated. PCBs, PAHs, chlorinated solvents, dioxins, furans, pesticides, hexachlorobenzene, BTXs, volatiles, and semi-volatiles have all been remediated. After treatment, the soils pass TCLP criteria for replacement or non-hazardous waste landfill disposal. Table 4 contains data from several PCB remediation projects.

Decontamination of Hexachlorobenzene-Contaminated Soil and Pesticide-Contaminated Soil

Na/NH₃ was very effective in destruction of hexachlorobenzene in soils. Sandy soil containing 67.6 ppm of hexachlorobenzene, from a site in Las Vegas, Nevada, was treated with Na/NH₃. Approximately 4% by wt. sodium was used. The treated soil contained <2 ppm of hexachlorobenzene. GC/MS analysis could detect no chlorinated products in the treated soil.

Pesticides in soils were also remediated. Table 5 summarizes some results from a project where soils from Hawaii and Virginia, contaminated with DDT, DDD, DDE and dieldrin, were treated with Na/NH₃. In all cases, the soils were remediated to non-detectable levels of the respective pesticide. Soils were shipped in from Naval facilities.

Treatment of Polyaromatic Hydrocarbons with Na/NH₃

Polyaromatic hydrocarbons (PAHs) are readily destroyed by solvated electrons in NH₃ both neat and in soils. Oligomeric reduced Birch reduction type products are obtained. These reactions are slower than dehalogenation as was demonstrated by comparing the rapid formation of benzene, toluene and naphthalene in Na/NH₃ from their corresponding monochloro derivatives versus rates of disappearance of PAHs under the same conditions. The PAHs in contaminated soils were remediated to below detection levels. The following compounds were remediated to almost nondetectable levels: acenaphthene, anthracene, benzo[a]anthracene, benzo[a]pyrene, pyrene, phenanthrene and fluorene.

Explosives

Most explosives are nitro or nitrate compounds. These functional groups are readily reduced by solvated electrons in NH₃. Aromatic nitro compounds may be eventually reduced to the corresponding amino derivatives. Azoxy-, azo- and hydrazoaryl dimers are obtained first. These dimers are eventually further reduced to the corresponding monomeric amino compounds. Experience is now being developed at both MSU and Commodore for detoxifying explosives including TNT, RDX, nitrocellulose, nitroglycerine, tetryl, PETN, Comp B, and M-28 with Na/NH₃.

Explosives have also been destroyed: (1) neat, (2) when mixed with the chemical warfare agents (3) from actual armaments and (4) in soils. No explosive analyte was found after treatment using EPA method 8330 (revision O, Sept. 1994). The reaction products were found to be oligomeric with all nitro groups reduced. Similar results were reported in Na/ethylenediamine, Ca/NH₃, and Ca/ethylenediamine where TNT and dinitrobenzene were destroyed (Mohammad 1996). Soils, collected from Los Alamos, New Mexico, contaminated with RDX, HMX and 1,2-dinitrobenzene, have been treated. After treatment with Na/NH₃, no detectable level of explosive was found (see Table 6).

Decontamination of Soils Containing Chlorooctane and 1,1,1-Trichloroethane

Standard B1 and B2 soil types, obtained from the Agronomy Department at MSU, were purposely contaminated with 3000 ppm of 1,1,1-trichloroethane and then treated with Na/NH₃. 1,1,1-Trichloroethane was selected since it is a widely dispersed pollutant in soils and DNAPLs. The quantity of Na required to lower the chlorocarbon level in the soil to a series of lower concentrations was determined on small aliquots of the contaminated soils. These results were expressed as the moles of Na consumed per mole of chlorine that was mineralized (e.g. RCl + nNa - NaCl + RH). Na consumption was then compared for dry versus wet soil samples to see the effect of water. In general, as the amount of RCl in the soil

decreases, the amount of Na required/Cl removed increases since other reactions compete more effectively at very low chlorocarbon concentrations.

While these studies were not taken to sub ppm levels of decontamination, lowering CH_2CCl_3 from 3000 to 1-8 ppm required only 5 equiv. of Na in dry B2 soil and only 22 equiv. of Na in the presence of 15% water (Table 7).

Similar data for tetrachloroethylene remediations are shown in Table 8 for B1 and B2 soils. Again, a higher water content causes more Na consumption. The overall Na requirements for the same levels of remediation are similar (on a per Cl atom basis) for dechlorination of CH_2CCl_3 and $\text{Cl}_2\text{C}=\text{CCl}_2$.

CONCLUSIONS

Solvated electrons react at diffusion controlled rates with CCl_4 , CH_2CCl_3 and chlorinated aromatic compounds under the conditions applied. The addition of substantial amounts of water only modestly increased the amount of Na needed to completely dechlorinate organic substrates. These diffusion-controlled reductions were complete in about 30 seconds, which was the time needed for complete metal dissolution in liquid ammonia. Diffusion control was supported by the absence of partially dechlorinated products in reductions of multiply chlorinated molecules in sodium-deficient partial reductions. Only the chlorinated phenols exhibited minor amounts of partially dechlorinated products in accord with a greater reluctance of the corresponding phenoxides to add an electron. The consumption of sodium was less than that of calcium, potassium and lithium, especially in the presence of water, suggesting sodium is the metal of choice for remediation.

Several types of PCB-, CAH-, pesticide- and explosive residue-containing wet soils were decontaminated in >99.9% efficiencies with Na/NH_3 (this includes clays, sandy soils and organic soils). Sample soils from superfund sites were successfully decontaminated to levels below 1.0 or 2.0 ppm. PCB-contaminated soils were cleaned using Na/NH_3 . The pesticides DDT, DDE and Dieldrin were remediated to levels <0.02 ppm in contaminated soils from two locations. Neat polynuclear aromatic hydrocarbons (PAHs) were destroyed in from 99.97 to 99.99% destruction efficiencies with single Na/NH_3 treatments. The

explosives HMX, RDX and 1,2-DNB were removed from Los Alamos, NM soil in removal efficiencies of 99.99 to 99.9999%.

Treating contaminated wet or dry B1 or B2 soils (containing, CH_2CCl_3 , $\text{Cl}_2\text{C}=\text{CCl}_2$ and $\text{Cl}_2\text{C}=\text{CHCl}$) with Na/NH_3 demonstrated each of these could be effectively remediated. The Na consumption required to reach specific contaminant levels, was studied, demonstrating that the Na consumed per Cl mineralized increased as the chloroorganic contaminants concentration remaining in the soil decreased. Wet soils required more Na to reach specified RCl concentrations than dry soils starting from the same concentration of RCl in the soil. However, the amount of sodium required did not increase excessively for wet soils.

EXPERIMENTAL METHODS

These have been recently described in detail (Pittman, He and Sun 2000).

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Table 1. Minimum Amount of Na Required to Completely Dechlorinate Chloroaromatics at 25°C

Entry	Substrate	Na/Substrate Mole Ratios and (Na/Per Cl Removed) Required For Complete Dechlorination at Different $\text{H}_2\text{O}/\text{Substrate}$ Mole Ratios			
		$\text{H}_2\text{O}/\text{Substrate}$			
		0/1 ^a	5/1	20/1	50/1
1	Chlorobenzene	1.5 (1.5)	2.3	2.4	2.5 (2.5)
2	4-Chloro-toluene	1.5 (1.5)	2.3	2.4	2.5 (2.5)
3	2-Chloro- <i>p</i> -xylene	1.5 (1.5)	2.3	2.4	2.5 (2.5)
4	1,2-Dichlorobenzene	2.8 (1.4)	4.0	4.5	5.0 (2.5)
5	3,4-Dichlorotoluene	2.5 (1.25)	4.2	4.6	6.0 (3)
6	1,2,3-Trichlorobenzene	4.0 (1.33)	5.5	5.8	7.0 (2.33)
7	1,2,3,4-Tetrachlorobenzene	5.0 (1.25)	6.5	7.0	8.6 (2.15)
8	4-Chlorophenol	2.0 (2)	2.9	4.0	4.5 (4.5)
9	2,4-Dichlorophenol	3.4 (1.7)	5.0	5.6	8.0 (4)
10	2,4,6-Trichlorophenol	4.6 (1.53)	7.4	8.0	10 (3.33)
11	1-Chlorooctane	1.7 (1.7)	2.3	2.5	4.0 (4)
12	1,1,1-Trichloroethane	3.6 (1.2)	4.2	4.5	5.1 (1.7)
13	Carbontetrachloride	4.6 (1.15)	5.1	5.5	6.4 (1.6)
14	Lindane	6.7 (1.11)	6.7	7.2	7.4 (1.23)

^a No water added. Moles of Na consumed per mole of chlorine removed, derived from Na/substrate weighings and divided by the number of chlorines in the substrate. Estimated errors $\pm 4\%$.

Table 2. Products Obtained in Sodium Deficient Reactions in Liquid NH₃ at room temperature ^a

Substrate	Na/Substrate (mole ratio)	H ₂ O/Substrate (mole ratio)	Products	
3,4-Dichloro- toluene	1	5	82% SM, ^b	18% toluene
	2	5	60% SM, ^b	40% toluene
	3	5	25% SM, ^b	75% toluene
1,2-Dichloro- benzene	3.5	5	7% SM, ^b	93% benzene
CCl ₄	1	0	79 % CCl ₄ ,	20 % CH ₄ ^c
	2	0	54 % CCl ₄ ,	45 % CH ₄ ^c
	3.4	0	33 % CCl ₄ ,	64 % CH ₄ ^c
CH ₃ CCl ₃	2	0	38% CH ₃ CCl ₃ , 62% CH ₃ CH ₃ ^d	

Table 3. Laboratory Treatments of PCB-Contaminated Soils with Ca/NH₃ at Room Temperature^a

Soil Matrix	Pre-treatment PCB Level (ppm)	Post-treatment PCB Level (ppm)	Destruction Efficiency (%)
Clay	290	0.05	>99.9
Clay	29	<0.06	>99.9
Sand	6,200	1.6	>99.9
Organic	660	0.16	>99.9
Organic	83	<0.04	>99.9

^a Experiments were carried out in a stirred 1.3L reactor made of steel. Prewedged soil samples were slurried for 10 to 20 min. in liquid NH₃ at ambient temperature. Then a calcium bar was dropped in. The calcium dissolved in a few seconds. The reduction reactions were completed as fast as the calcium dissolved.

Table 4. Destruction of PCBs in Various Soils with Na/NH₃

Source of Soil	Soil type	Pre-Treatment PCB Level (ppm)	Post-Treatment PCB Level (ppm)
Harrisburg, PA ^a	Sand, clay	777	<1.0
Los Alamos, NM ^b	Sand, silt, clay	77	<2.0
New York ^c	Sand, silt	1250	<2.0
Monroe, LA ^d	Sand, silt, clay	8.8	<1.0

Treatment temperature in liquid NH₃ a) 32°C, b) 20°C, c) -33°C, d) -33°C. The range of Na wt. percents in NH₃ used was 1.27 to 3.3% in these examples.

Table 5. Destruction of Pesticides in Soil with Na/NH₃

	DDT	DDT	DDE	Dieldrin
Barbers Point, HI				
Pre-treatment	200	180	69	ND
Post-treatment	<.02	<.02	<.02	ND
Dahlgren, VA				
Pre-treatment	9	1.6	ND	15
Post-treatment	<.02	<.02	ND	<.02

ND = Not Detected. Other values in parts per million.

Table 6. Destruction of Explosives in Soil From Los Alamos, New Mexico Using Na/NH₃.^a

	HMX	RDX	1,2 DNB
Soil Before Treatment (mg/kg)	1600	3580	9.6
Treated (mg/kg)	.03	.03	.03
Detection Limit	.03	.03	.03
Destruction Efficiency (%)	99.9999	99.99999	99.99

^a Na (2.8 wt%) in NH₃ (1L) used per 50g of soil at 39°C.

Table 7. Remediation of CH_3CCl_3 in B1 (Clay) and B2 (Organic) Soils. Sodium Consumption per Chlorine Mineralized at Different Levels of Water and Using Different Amounts of NH_3 .

Entry No.	Soil Type and Water Content	NH_3 vol. g. soil	CH_3CCl_3 Level in Soil (ppm)		Na Consumed per Cl Removed
			Start	Finish	
1	B1 Dry	3	3000	9.6	7.4
2		5	3000	8.0	7.4
3		10	3000	7.5	7.4
4	B1 7% H_2O	3	3000	133	15
5		10	3000	131	15
6		3	3000	9.5	22
7		10	3000	2.2	22
8	B1 15% H_2O	5	3000	381	22
9		10	3000	361	22
10	B2 Dry	5	3000	1.7	5
11	B2 7% H_2O	5	3000	7.2	22
12	B2 15% H_2O	5	3000	4.1	22

^a Cumulated over the drop of CH_3CCl_3 concentrations shown in each example. The same amount of Na was added to entries 1-3 but that amount was different than that added to entries 4 and 5. Entries 6-9 and 11-12 all used the same amount of Na (but that amount was different than the other runs). All reactions were run until all the sodium was consumed.

Table 8. Remediation of Tetrachloroethylene in B1 and B2 Soils. Sodium Consumption Per Chlorine Mineralized Versus Water Content of Soil.

Soil Type / Water Content	$\text{Cl}_2\text{C}=\text{CCl}_2$ level in Soil (ppm)		Na Consumed per Cl Removed
	Start	Finish	
B1 Dry	3000	0.9	9
B1 7% H_2O	3000	0.8	26
B1 15% H_2O	3000	2.0	35
B2 Dry	3000	4.0	9
B2 7% H_2O	3000	4.6	21
B2 15% H_2O	3000	2.6	27