Sustainable Remediation of Vadose Zone Soils Contaminated with Recalcitrant Organics and Heavy Metals

Webinar

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Agenda

Treatment Approach

- in-situ, ex-situ, deep soil mixing
- Eliminate Transportation & Off-site Disposal
- Sustainability Comparison

Evonik Reagent Formulations

- Terramend[®] for Enhanced Aerobic Bioremediation
- Daramend[®] for In Situ Chemical Reduction
- MetaFix[®] for Removal of Dissolved Metals
- Microbiology, Biochemistry, and Chemistry

Bench-scale Treatability Testing

- Benefits
- Methodology
- Representative Results

Project Snapshots

- Terramend[®] (PAHs, PCP, phthalates)
- Daramend[®] (Chlorinated Pesticides, Organic Explosives)
- MetaFix[®] (Lead, Mercury)

Questions & Answers





In Situ Treatment of Chlorinated Pesticides

Former Agricultural Site to Residential Development





Ex Situ Treatment of Chlorinated Phenols and cVOCs Chemical Manufacturing Site



Increasing consideration of sustainability highlights the benefits associated with in situ and on-site remediation of vadose zone soils.



On Site Windrow Treatment of Organic Explosive-impacted Soil US Army Site





In Situ Daramend[®] Treatment of Organic Trinitrotoluene US Navy Site: Deep soil mixing from 0 - 8' bgs





Sustainability Comparison

Estimates based on data from completed project in 2018

Task	In-Situ Daramend® Treatment	Excavation & Landfill Disposal		
	Estimated CO ₂ Emissions (tons)			
Excavation	-	248		
Trucking to Landfill	-	1,145		
Tillage	185	-		
Irrigation	25	-		
Daramend [®] Production	45	-		
Daramend [®] Trucking	10	-		
Other Site Work	10	5		
Total	275	1,398		

Notes:

1. Excavation estimate based on 50 y³/hour and total yardage of 112,770 resulting in total excavator hours of 2,255.

2. Trucking estimate based on 36 miles to landfill, standard 6-cylinder diesel tandem-axle dump, 13 y3/load (i.e., 8,675 loads), empty return trip.

3. Tillage and irrigation estimates based on project records (34-acre site, 1.0 acre/day, 4 treatment cycles, treatment depth was 0 – 24" bgs).

4. Other site activities include travel to/from site including project staff (three people; 5 days/week; 16 weeks) and consultant (1 person, 2 days/week; 16 weeks).

5. Daramend[®] production estimate based in internal data from production in Canada.

6. Daramend[®] trucking assumes 70 miles to facility, 560 tons delivered, 27 loads at 21 tons per load, empty return trip.



Sustainable Remediation of Soil in the Vadose Zone

Treatment Technologies Reviewed

Terramend® Enhanced Aerobic Bioremediation

- PAHs, PCP, phthalates, petroleum hydrocarbons
- Some chlorinated herbicides and pesticides

Daramend[®] In Situ Chemical Reduction

- Chlorinated pesticides and herbicides
- Organic explosive compounds
- Chlorinated VOCs

MetaFix[®] Removal of Dissolved Metals

- Heavy metals
- Metalloids
- Organometallic compounds



Enhanced Aerobic Bioremediation

- 1. Perspective on Aerobic Bioremediation
- 2. Terramend[®] Reagents
- 3. Treatment Mechanisms
- 4. Bench-scale Test Results
- 5. Project Snapshots



Generic Approach to Aerobic Soil Bioremediation

- Traditional focus has been on supplying adequate inorganic N & P to support biodegradation of target hydrocarbons, adjusting soil water content, and soil mixing for aeration.
- Generally supplied in the form of commercial/agricultural fertilizer
- Target an "optimized" C:N:P ratio based on an estimate of bioavailable carbon including target compounds and native organic matter
- Commonly target C:N:P at 120:10:1 molar ratio
- This approach can fail because inorganic nutrients are used very quickly when bioavailable and are also lost through wasteful processes including luxury consumption, <u>rapid denitrification</u>, and precipitation as <u>insoluble aluminum</u>, iron, and calcium phosphates.
- This approach also fails to address the issue of low bioavailability of water in hydrophobic soils and the acute microbial toxicity created by some contaminants (e.g., PCP, Lindane)
- We use a different approach that is based on supplying nutrients, increasing bioavailable water, and overcoming acute microbial toxicity with our Terramend[®] family of soil amendments.

Nutrient Loss Mechanisms

Denitrification

 $5(CH_2O) + 4NO_3^- + 4H^+ \rightarrow 5CO_2 + 2N_2^+ + 7H_2O$

Can be rapid, complete loss in 2 – 3 days in wet soil

Phosphate Precipitation

 $AI(OH)_2^+ + H_2PO_4^- \rightarrow AI(OH)_2H_2PO_4 \downarrow$

- Dominant reaction under acidic conditions
- Various iron phosphates also precipitate

 $3Ca^{2+} + 2PO_4^{3-} \rightarrow Ca_3(PO_4)_2$

• Dominant reaction under alkaline conditions



Terramend® Reagent Formulations

Attribute	Terramend [®] Carbon	Terramend [®] Inorganic	Benefits
High Surface Area Hydrophilic Plant Fiber	✓	\checkmark	Large increase in soil water holding capacity (>50%) enables development of larger more vigorous microbial population, reduces acute microbial toxicity
Slow-release Organic Carbon & Nutrients (N, P, K, S)	\checkmark	\checkmark	Provides continuous supply of biodegradable nutrients to support microbial growth. Reduces treatment cost by elimination need for repeated nutrient applications
Rapid-release Organic Carbon & Nutrients (N, P, K, S)	-	\checkmark	Enables quick start to contaminant degradation
Inorganic Nitrogen	-	\checkmark	Beneficial when initial hydrocarbon content is very high.
Inorganic Phosphorus	-	\checkmark	Beneficial when initial hydrocarbon content is very high.
Emulsifying Agent	\checkmark	\checkmark	Increases desorption of contaminants with low water solubility
pH Buffer	\checkmark	\checkmark	Prevents acidification sometimes observed when high concentrations of chlorinated compounds are treated



Rapid & Slow-Release Organic Carbon, Nitrogen & Phosphorus in Terramend[®] Reagents



Carbon & Nutrients in Terramend® Reagents

- Between 4% and 10% in rapidly available forms including sugars, polysaccharides, and amino acids.
- Remainder is in more slowly-released forms including cellulose, hemicellulose, and amino acid-oligosaccharide structures
- Complex organic structures are hydrolyzed by one subgroup of soil microbial population to produce simpler molecules that can be used by many other subgroups
- Provides a range of rapidly and slowly released carbon and nutrients to support a variety of aerobic bioremediation applications
- Metabolism of these compounds results in stable, supply of nitrogen and phosphorus for bacteria and fungi important to effective degradation of hydrocarbons
- Natural, sustainable, long-lasting food source for both bacteria and fungi in soil



Influence of Terramend[®] Dosage Soil Water Holding Capacity

Sandy Loam Soil (Total PAH = 1,710 mg/kg, Total CPs = 352 mg/kg, TPH = 7,300 mg/kg)





Bench-scale Treatability Testing

Objectives

- ✓ Confirm adequate removal and treatment time
- ✓ Determine effective reagent dosage
- ✓ Establish fate of target compounds

Methodology

- ✓ Representative soil
- ✓ Conservative conditions
- ✓ Replication

Representative Results

- ✓ PCP
- ✓ TPH



Bench-scale Treatability and Fate Testing for PCP in Soil Mineralization of ¹⁴C-PCP to ¹⁴C-CO₂





Bench-scale Treatability Testing for PCP in Soil Total Extractable Chlorinated Phenols



Initial ■ 81 days ■ 151 days

- Very good agreement between mineralization of ¹⁴C-PCP and reduction in total extractable CPs
- ✓ Treatments that supported greatest conversion of radiolabeled PCP to CO₂ also achieved lowest residual CP concentrations
- Higher dose of slowly-released Terramend[®] Carbon provided large increase in soil WHC without turning soil anaerobic
- More rapidly-released Terramend[®] Inorganic at the higher dose may have resulted in less oxic conditions in this soil
- ✓ Inorganic N+P (fertilizer) was ineffective regardless of dosage



Bench-scale Treatability Testing

Terramend[®] Treatment of Stabilized Drill Cuttings



- Very high TPH drill cuttings from oil extraction
- Initial treatment was stabilization with wood mulch followed by aerobic tillage; however, this approach did not achieve the desired degree of TPH reduction
- The as-received drill cuttings were subjected to 141 days of Terramend[®] treatment at RT with weekly aeration by mixing
- Sharp difference in response to Terramend[®] Inorganic vs Terramend[®] Carbon.
- Most effective treatment was the higher dosage of Terramend[®] Inorganic.
- Very high demand for nitrogen and phosphorus due to high TPH and additional organic carbon from wood mulch
- TPH was reduced by 91% in response to this treatment



Terramend® Project Snapshot 1

Terramend[®] Carbon On-Site Treatment of Phthalates in Excavated Soil

- Site in eastern New Jersey
- Former chemical industry facility
- Approximately 600 tons of soil treated on-site
- Two 300-ton batches in HDPElined biocell





Bench-scale Treatability Testing Results

Bis(2-ethylhexyl) phthalate in New Jersey Chemical Industry Soil



■Initial ■ 30 days ■ 130 days

Interpretation of Results

- Low solubility of phthalate makes it relatively slow to degrade
- Terramend[®] Inorganic at 2.0% w/w and Terramend[®] Carbon at 3.0% w/w.
- Longer lasting reagent (Terramend[®] Carbon) was better suited
- High native organic matter and fine-grained texture made it more difficult to maintain aerobic conditions in soil treated with Terramend[®] Inorganic
- Possible that native bacteria produced more biosurfactant under lower nutrient status in soil with Terramend[®] Carbon
- Visible fungal growth on soil with Terramend[®] Carbon



Terramend® Inorganic Treatment of Bis(2-ethylhexyl) Phthalate

Ex situ, On-Site Treatment, New Jersey



- Ex situ soil treatment on-site in NW New Jersey
- Terramend[®] formulation and dosage established during bench-scale treatability testing
- Soil moisture set to 60% WHC by spray irrigation system
- Reagent dosage of 3.0% w/w
- Slowly released carbon and nutrients
 was proven more effective
- Visible fungal growth on soil treated with Terramend[®] Carbon
- Treatment through winter months so active treatment was about 100 days.



Project Snapshot 2

Terramend[®] Carbon

Ex Situ Treatment of PAHs, PCP, and Petroleum Hydrocarbons

- Industrial Wood Preserving Site
- In Situ treatment of soil from 0 24" bgs (4,800 tons)
- On-site treatment of excavated soil in HDPE-lined cell
- 1,200 tons/year in batch system.







Ex Situ Bioremediation of Wood Treatment Soil with Terramend® Carbon

Influence of Terramend® reagent on total chlorinated phenols



- Industrial wood preserving site in operation since 1950
- Pressure treatment using creosote and PCP in mineral oil
- Batch treatment of 1,200 tons/year over three years
- Excavated soil in HDPE-lined bioremediation cell
- Covered to extend treatment season in cool climate area
- First batch included monitoring of untreated control soil simultaneous with Terramend[®] Carbon treated soil
- Also treated 4,800 tons of lightly impacted soil in-situ (0 – 24" bgs)
- Terramend dosage was 3.0% 4.5% (ex-situ) and 1.5% - 2.5% (in-situ)



Ex Situ Bioremediation of Wood Treatment Soil with Terramend® Carbon

Influence of Terramend[®] reagent on total PAH and TPH concentrations





In-Situ Chemical Reduction

Anaerobic Bioremediation with Organic Carbon + ZVI Reagents

- 1. Daramend[®] Reagents
- 2. Treatment Mechanisms
- 3. Bench-scale Test Results
- 4. Project Snapshots



Daramend® Reagent Formulations

Attribute	Daramend®	Daramend [®] Plus	Benefits
High Surface Area Hydrophilic Plant Fiber	\checkmark	\checkmark	↑ Soil WHC, New non-toxic surface area, long- lasting source of VFAs
Slow-release Organic Carbon & Nutrients (N, P, S)	\checkmark	\checkmark	Feed bacteria without inhibition of fungi by avoiding excess soluble nitrogen, organosulfur compounds
Rapid-release Organic Carbon	\checkmark	\checkmark	Rapid creation of strong reducing conditions to promote reductive dehalogenation/degradation
Emulsifying Agent	\checkmark	\checkmark	Increase bioavailability of contaminants
pH Balanced	\checkmark	\checkmark	Prevent acidification by balancing acidity from VFAs
Microscale ZVI (Fe ⁰)	\checkmark	\checkmark	Direct dechlorination by surface contact, long- lasting source of Fe ²⁺ , biogenic reactive minerals
Activated Carbon	-	\checkmark	Physical adsorption of pesticides to rapid pass of TCLP



Daramend[®] Treatment Mechanisms

Carbon Fermentation + ZVI Corrosion: Synergy Promotes Multiple Dechlorination Mechanisms



Favorable thermodynamic conditions for dechlorination:

- Combined oxygen consumption from carbon fermentation and iron oxidation \rightarrow Strongly reduced environment (-250 to -500 mV)
- High electron/H⁺ pressure

$Fe^0 \rightarrow Fe^{2+} + 2e^{-}$ $2H_2O \rightarrow 2H^+ + 2OH^ 2H^+ + 2e^- \rightarrow H_{2(g)}$ $R-CI + H^+ + 2e^- \rightarrow R-H + CI^-$

Production of organic acids (VFAs):

- Serves as electron donor for microbial reduction of cVOCs and other oxidized species
- The release of acids keeps the pH down and thereby serve to reduce precipitate formation on ZVI surfaces to increase reactivity
- Increase rate of iron corrosion/H₂ generation



Bench-scale Treatability Testing

Objectives

- ✓ Confirm adequate removal and treatment time
- ✓ Determine effective reagent dosage
- Establish fate of target compounds

Methodology

- ✓ Representative soil
- Conservative conditions
- ✓ Replication

Representative Results

- ✓ Chlorinated pesticides
- ✓ Dieldrin, Aldrin, Chlordane, Toxaphene



Daramend® Bench Scale Results

Treatment of Aldrin, Lindane, Chlordane, and Dieldrin in Louisiana soil

Chlorinated Pesticide Concentrations after Daramend® Treatment



Aldrin Lindane Chlordane Dieldrin

Aldrin, Lindane, Chlordane, and Dieldrin are some of the most recalcitrant chlorinated pesticides



Daramend® Bench Scale Results:

Influence on reagent dosage on total pesticide concentration in Florida soil



Notes:

1. Data points represent mean of three replicates.

2. Dosage values are per treatment cycle.

Daramend[®] Dosage Response

- Example of relationship between Daramend[®] dosage and Chlordane + Dieldrin degradation
- Four treatment cycles completed over a period of 36 days
- Results enable consultant and site owner to select reagent dosage in view of reagent cost and available time for completion of treatment.



Daramend[®] Bench Scale Results:

Removal of Toxaphene by treatment cycle (Alabama soil)



Influence of Daramend[®] Treatment on Toxaphene Concentrations

Daramend® Treatment of Toxaphene

- Example of relationship between number of cycles and pesticide removal
- A treatment cycle involves addition of Daramend[®], soil mixing, and irrigation.
- Depends on soil characteristics, temperature, contaminant chemistry and concentration
- Once reductive conditions are established contaminant removal response increases significantly
- Mixing is optional for treatment of OE (higher water solubility)



Days (cycles)

Data points represent mean of four replicates; $LSD_{(0.05)} = 6.5$

Project Snapshot 1

Daramend®

In Situ Treatment of Chlorinated Pesticides

- Former agricultural site (apple orchard and strawberry fields)
- Conversion to residential development
- In situ treatment of 34 acres (14 hectares)
- About 110,000 tons of soil in total
- Surface soil to a depth of 24" (60 cm)
- Residual metabolites from use of DDT as an insecticide
- Major target compound was DDE







In Situ Daramend® Treatment of DDE Contaminated Soil

Former Agricultural Site Remediation for Residential Development



- Daramend[®] reagent dosage was 0.15% w/w per treatment cycle
- Each treatment cycle includes reagent application, tillage, and irrigation
- Pesticide analysis by SW-846 Methods 3541 (automated Soxhlet) and 8081B (GC ECD)



Achieved Residential Criteria

- Treatment of large areas in situ sharply reduces total cost
- Total cost including Daramend[®], equipment, and labor was less than US\$50,000/acre
- This is equivalent to <\$20/ton of treated soil



Daramend[®] Project Snapshot 2 Ex-situ Treatment of TNT in Soil Naval Weapons Station Yorktown Yorktown VA

- 8,400 y³ soil (*ca.* 12,000 tons)
- Soil TNT concentrations as high as 43,000 mg/kg (average about 10,000 mg/kg)
- Treatment goals were 14 mg/kg for TNT and 5 mg/kg for RDX
- Daramend[®] was selected through the FS Process
- Ex Situ treatment of soil and sediment (impacted by effluent from washout of TNT manufacturing plant)
- Engineered HDPE biocell, covered to prevent flooding and allow extended treatment season







Daramend® Project Snapshot 2

Naval Weapons Station Yorktown VA





Daramend® Project Snapshot 2

TNT Concentrations before and after Daramend® Treatment

	Bato	:h #1	Batc	h #2	Batc	h #3	Batc	h #4	
Samplin g Zone	Initial	Final	Initial	Final	Initial	Final	Initial	Final	
9 _00		TNT (mg/kg)							
1	14,000	4.1	240	4.0	1,520	0.6	12,400	2.0	
2	7,900	6.5	3,500	5.6	2,400	10.4	5,700	12.0	
3	12,000	3.1	1,600	7.1	1,560	0.5	43,400	2.4	
4	17,000	7.0	38,650	3.6	8,000	1.0	351	1.3	
5	19.0	2.6	7,000	0.3	2,210	2.7	929	1.3	
6	5,100	5.7	5,900	3.3	15,500	11.5	192	1.0	
7	33,000	8.8	9,300	1.8	30,200	5.7	19.5	1.2	
8	1,300	2.9	31,873	1.2	10,900	2.0	5,870	1.1	
9	8,400	14.0	1,000	14.0	40,400	9.5	333	0.8	
10	2,800	6.8	1.7	4.0	40,900	8.8	12,000	12.6	
Mean	10,151	6.2	9,906	4.5	15,359	5.3	8,119	3.6	

Treatment Protocol

- ✓ Daramend dosage of 1.0%
 w/w for first cycle and 0.5%
 w/w for subsequent cycles
- ✓ Each treatment cycle includes addition of Daramend, tillage, irrigation, and a post irrigation reaction period of 7 – 10 days
- ✓ Soil moisture content set to 70% to 80% of WHC
- Lower soil moisture content for treatment of organic explosive compounds than for chlorinated pesticides



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Metals Treatment

Removal of Dissolved Heavy Metals and Metalloids

- 1. MetaFix[®] and GeoForm[®] Reagents
- 2. Treatment Mechanisms
- 3. Bench-scale Test Results
- 4. Project Snapshots





MetaFix® and GeoForm® Reagent Formulations

Attribute	MetaFix [®] I-3	MetaFix® I-6A	MetaFix® I-7A	GeoForm® ER
ZVI	\checkmark	\checkmark	\checkmark	
Iron Sulfide	\checkmark	\checkmark	\checkmark	
Iron Oxides	\checkmark	\checkmark	\checkmark	✓
Carbonate	-	\checkmark	-	
Activated Carbon	-	-	\checkmark	
Slow-release organic carbon and nutrients	-	-	-	\checkmark
Quick-release soluble organic carbon	-	-	-	\checkmark
Ferrous Iron and Sulfate	-	-	-	\checkmark
pH Buffer	-	-	-	\checkmark



Removal of Dissolved Metals

Metal Sulfides vs Metal Hydroxides (Lower Solubility & Broader pH Range Stability)



EPA 625/8-80-003, 1980; Banerjee et al., 2013. Veolia Water Inc. Environ. Sci. Technol. 1988, 22, 972-977



Sulfate-Reducing Bacteria (SRB)

In Situ Generation of Sulfide for Precipitation of Dissolved Metals with GeoForm®

- Anaerobic bacteria that use inorganic sulfur compounds (i.e., sulfate, sulfite, thiosulfate) as their terminal electron acceptor during anaerobic respiration and produce sulfide (S²⁻, HS⁻, H₂S).
- This is a large and diverse group of bacteria (40 genera, >120 species).
- Free sulfide promotes corrosion of iron and steel (bad for life of UST, but good for flow of Fe²⁺ from ZVI).
- Require simple organic electron donors (VFAs, ethanol, glucose).
- Steady flow of fermentation products and phosphate from metabolism of plant fiber supports growth and stability of SRB population
- It is important that sulfate must be activated by the enzyme ATP sulfurylase before reduction to sulfide can begin (*n.b.*, importance of bioavailable phosphate, reagent considerations).



Figure 1. Mixed bacterial biofilm on iron particle.





MetaFix® Project Snapshot 1

Lead-impacted, industrial process waste

- Sand blasting residue
- Strongly acidic with high TCLP lead
- Direct soil mixing with excavator
- MetaFix[®] I-6A reagent dosage at 6.0 % w/w
- Soil water content set to 80% of WHC (wet, but not saturated)
- Treatment time was 7 days
- Earlier attempts at treatment with FeSO₄ and fly ash at 40% w/w were ineffective
- TCLP lead was reduced from 11.7 mg/L to 0.22 mg/L





MetaFix® Project Snapshot 2

On-Site Treatment of Mercury: Bench-scale Treatability Test



- Mercuric chloride was used as a catalyst in chemical synthesis at this former chemical plant
- Soil mercury concentrations ranged from 300 to 420 mg/kg.
- Results from bench-scale testing indicated that a MetaFix® dosage of 0.5% (w/w) would achieve the remedial objective for mercury (i.e., 1.0 µg/L SPLP).

Parameter	Control (untreated)	MetaFix® 0.5% (w/w)	MetaFix® 1.0% (w/w)
Soil Moisture (%)	18.3	18.5	20.0
рН	8.6	8.0	7.9
Total Hg (mg/kg)	315	293	314
SPLP Hg (µg/L)	35.1	<1.0	<1.0



MetaFix[®] Project Snapshot 2 On-Site Treatment of Mercury: Pilot-scale Demonstration





Pilot-scale Demonstration

- Four treatment cells, each with 50 m3 soil
- A range of MetaFix® dosages compared including 0.5% 1.0%, and 2.0% (w/w)
- MetaFix reagent mixed into the soil with an excavator bucket and a screening bucket
- Sufficient water added to increase soil moisture content to near but below the point of saturation (ca. 90% WHC)
- Soil was covered with HDPE to prevent drying and support creation of anaerobic conditions
- Reaction period was 7 days
- Even the lowest dosage achieve the remedial objective of 1.0 µg Hg/L in SPLP testing
- Decision was to use 0.5% w/w reagent dosage for fullscale application



Sustainable Remediation of Vadose Zone Soil

MetaFix[®] Project Snapshot 2 On-Site Treatment of Mercury: Full-scale Treatment

Allu Screening Bucket



Full-scale Application

- The MetaFix[®] dosage of 0.5% w/w used for the full-scale treatment.
- Full scale implementation utilized an integrated soil mixing system where soil crushing/screening and reagent dosing/mixing are completed in a single process.
- Reaction period was the same as in the pilot-scale demonstration (7 days).
- Several thousand tons successfully treated to the SPLP remedial goal.
- Achieved mercury remedial standard (i.e., <1.0 µg/L SPLP</p>



Summary

Terramend® Enhanced Aerobic Bioremediation

- 30-year record of success on TPH, PAHs, PCP, and phthalates
- Improves treatment kinetics by increasing bioavailable water
- Eliminates need for repeated applications of nutrients in fertilizer forms
- Enables treatment of even heavily contaminated, hydrophobic soils

Daramend[®] In Situ Chemical Reduction for OCPs & OE

- 25-year record of success on chlorinated pesticides, herbicides, and organic explosive compounds
- Effective on even the most recalcitrant OCPs including Dieldrin, Chlordane, Toxaphene, and Lindane
- Reduce costs associated with excavation, transportation, and landfilling of soils classed as hazardous
- Cost factor can be as little as 1/3 of "dig & dump" approach

MetaFix[®] Removal of Dissolved Metals and Metalloids

- Remove dissolved metals in situ or on site to avoid excavation, transportation, and landfill costs
- Converts dissolved metals into low solubility mineral forms by sulfide precipitation or iron coprecipitation
- Precipitates have lower solubility and broader pH range stability than alkaline treatment methods



Questions are Welcome!



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