Testing and Recommended Practices to Improve Nurse Tank Safety: Phase II



U.S. Department of Transportation Federal Motor Carrier Safety Administration

December 2013

FOREWORD

This is the final report for Phase II in a multi-year research project examining options for, and practicality of, non-destructive examination (NDE) of nurse tanks, which are used to transport anhydrous ammonia (NH₃) locally from retailers over public roadways and farm fields. Historically the tanks were approximately 1,000 or 1,500 gallons; however, some individuals (more recently) are using dual and tri-mounted tank running gear. On the road, nurse tanks can be pulled by pickup trucks or tractors. They are sometimes involved in incidents such as overturning, which can damage their valves and cause them to vent ammonia.

Nurse tanks hold NH_3 at multiple times the atmospheric pressure. NH_3 is a hazardous material that can cause chemical burns, frostbite, and suffocation. Tank failures can release this pressure with catastrophic force, posing the risk of impact injury to workers and bystanders.

This report presents findings from the following:

- Examination of causes for pinhole leaks and risk of failure.
- Extended testing of stress corrosion crack (SCC) growth measurement.
- Comparison of residual stress in annealed versus unannealed nurse tanks.
- Statistical findings of indications in a sample of 532 in-service nurse tanks both with and without annealing.

This document reports the findings and recommendations for best inspection practices to reduce risks associated with nurse tank failures.

NOTICE

This document is disseminated under the sponsorship of the U.S. Department of Transportation (USDOT) in the interest of information exchange. The U. S. Government assumes no liability for the use of the information contained in this document. The contents of this report reflect the views of the contractor, who is responsible for the accuracy of the data presented herein. The contents do not necessarily reflect the official policy of the USDOT. This report does not constitute a standard, specification, or regulation.

The U. S. Government does not endorse products or manufacturers named herein. Trademarks or manufacturers' names appear in this report only because they are considered essential to the objective of this report.

QUALITY ASSURANCE STATEMENT

The Federal Motor Carrier Safety Administration (FMCSA) provides high-quality information to serve Government, industry, and the public in a manner that promotes public understanding. Standards and policies are used to ensure and maximize the quality, objectivity, utility, and integrity of its information. FMCSA periodically reviews quality issues and adjusts its programs and processes to ensure continuous quality improvement.

Technical Report Documentation Page

1. Report No. FMCSA-RRR-13-055	2. Government Accession N	o. <u>3</u> .	Recipient's Catalog No.		
4. Title and Subtitle Testing and Recommended Practices to Improve Nurse Tank S Phase II		Sank Safety:5.D	Report Date ecember 2013		
		6.	Performing Organization C	Code	
7. Author(s) Russell, Alan; Chumbley, Scott;	Becker, Andrew	8.	Performing Organization F	Report No.	
9. Performing Organization Name and Add Iowa State University	ress	10	. Work Unit No. (TRAIS)		
2220K Hoover Hall Ames, IA 50011		11	. Contract or Grant No.		
12. Sponsoring Agency Name and Address U.S. Department of Transportati Federal Motor Carrier Safety Ad	on Iministration and Pipelin	ne and 20	. Type of Report and Perio nal Report, October 013	d Covered •, 2010–August,	
Hazardous Materials Administration Office of Analysis, Research, and Technology 1200 New Jersey Ave. SE Washington DC 20590			14. Sponsoring Agency Code FMCSA		
15. Supplementary Notes Contracting Officer's Represent:	ative: David Goettee				
 16. Abstract This project addressed four topics: Pinhole leaks in nurse tanks were studied by radiography, serial milling, and side-angle ultrasound. These measurements indicated that welding surfaces contaminated by water, mill scale, rust, or other contaminants caused weld porosity that caused greatly accelerated metal fatigue of remaining non-porous wall thicknesses, which rapidly led to complete tank wall penetration, causing slow leakage of NH₃. Stress corrosion crack (SCC) growth rates were measured in three different ammonia solutions. These tests showed that neither nitrogen purging of a tank's vapor space during filling nor adding N-Serve to ammonia changed crack growth rates. Neutron diffraction analysis on a nurse tank given an American Society of Mechanical Engineers (ASME) protocol post-weld heat treatment (PWHT) lowered residual hoop stress near welds by two-thirds and lowered residual axial stress near welds by one-third. Since residual stress drives initiation and propagation of stress corrosion cracking, these findings suggest that SCC should be greatly reduced by PWHT in tank manufacture. Side-angle ultrasound measurements of welds in 532 in-service nurse tanks showed that 40 percent of the tanks had no observable cracks, and the remaining 60 percent of tanks contained a total of 3,326 indications. Cracks are most common in tank heads near the circumferential welds in the vapor space near the tanks with greater porosity in their welds appear to have more indications. 					
17. Key Words Hazardous material, anhydrous a	ammonia, nurse tanks	18. Distribution Stateme No restrictions	nt		
19. Security Classif. (of this report) Unclassified	20. Security Classif. (c Unclassified	f this page)	21. No. of Pages 115	22. Price	

Form DOT F 1700.7 (8-72)

Reproduction of completed page authorized.

	SI" (INIODERIN IN	EIRIC) CONVER	SION FACIORS	
	TABLE OF AP	PROXIMATE CONVERSION	NS TO SI UNITS	
Symbol	When You Know	Multiply By	To Find	Symbol
·	to all a s	LENGTH	NATION ALCON	
in #	Inches	25.4	Maters	mm
π	feet	0.305	Meters	m
yd	yards	0.914	Meters	m
mi	miles	1.61	Kilometers	km
		AREA		
in ²	square inches	645.2	square millimeters	mm²
ft ²	square feet	0.093	square meters	m²
yd²	square yards	0.836	square meters	m²
ac	acres	0.405	Hectares	ha
mi²	square miles	2.59	square kilometers	km²
		VOLUME	1,000 L shall be shown in m ³	
fl oz	fluid ounces	29.57	Milliliters	mL
gal	gallons	3.785	Liters	L
ft ³	cubic feet	0.028	cubic meters	m³
yd ³	cubic yards	0.765	cubic meters	m³
	,	MASS		
oz	ounces	28.35	Grams	a
lb	pounds	0 454	Kilograms	ka
Т	short tons (2000 lb)	0.907	megagrams (or "metric ton")	Ma (or "t")
	31011 (2000 15)	TEMDEDATURE	Tomporaturo is in oxact dogrado	ing (or t)
∘⊏	Fabranhait	$5 \times (E 22) \cdot 0$	Coloine	°C
F	ramennen	$5 \times (1-52) \div 5$	Celsius	C
		01 (F-32) ÷ 1.6		
		ILLUMINATION		
fC	foot-candles	10.76	Lux	IX
tl	foot-Lamberts	3.426	candela/m ²	cd/m ²
		Force and Pressure or Stress		
16+	noundtoroo	a ai	Noutono	N
	poundiorce	4.45	Newtons	
lbf/in²	poundforce per square inch	4.45 6.89	Kilopascals	kPa
lbf/in ²	poundforce per square inch	4.45 6.89 ROXIMATE CONVERSIONS	Kilopascals	kPa
Ibi Ibf/in ²	poundforce per square inch TABLE OF APP When You Know	4.45 6.89 ROXIMATE CONVERSIONS Multiply By	Kilopascals S FROM SI UNITS To Find	kPa Symbol
Ibf/in ² Symbol	poundforce per square inch TABLE OF APP When You Know	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH	Kilopascals S FROM SI UNITS To Find	kPa Symbol
Symbol	poundforce per square inch TABLE OF APP When You Know millimeters	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039	Kilopascals S FROM SI UNITS To Find inches	kPa Symbol
Symbol Mm	poundforce per square inch TABLE OF APP When You Know millimeters meters	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28	FROM SI UNITS To Find inches feet	kPa Symbol in ft
Symbol Mm M m	poundforce per square inch TABLE OF APP When You Know millimeters meters meters meters	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09	FROM SI UNITS Fro Find inches feet yards	kPa Symbol in ft yd
Ibl Ibf/in ² Symbol Mm M M m km	poundforce per square inch TABLE OF APP When You Know millimeters meters meters kilometers	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621	inches feet yards miles	kPa Symbol in ft yd mi
Symbol Mm M m km	poundforce per square inch TABLE OF APP When You Know millimeters meters meters kilometers	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA	FROM SI UNITS FROM SI UNITS To Find inches feet yards miles	kPa Symbol in ft yd mi
Symbol Mm M m km mm ²	poundforce per square inch TABLE OF APP When You Know millimeters meters meters kilometers square millimeters	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016	Kewtons Kilopascals FROM SI UNITS To Find inches feet yards miles square inches	kPa Symbol in ft yd mi in ²
Mm Mm km m2 m2	poundforce per square inch TABLE OF APP When You Know millimeters meters meters kilometers square millimeters square meters	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764	Kewtons Kilopascals S FROM SI UNITS To Find inches feet yards miles square inches square feet	kPa Symbol in ft yd mi in ² ft ²
Ibl Ibf/in ² Symbol Mm M M m km km m ² m ²	poundforce per square inch TABLE OF APP When You Know millimeters meters meters kilometers square millimeters square meters square meters square meters	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195	Kewtons Kilopascals FROM SI UNITS To Find inches feet yards miles square inches square feet square yards	kPa Symbol in ft yd mi in ² ft ² yd ²
Ibl Ibf/in ² Symbol Mm M M M Km km km m ² m ² ha	poundforce per square inch TABLE OF APP When You Know millimeters meters meters kilometers square millimeters square meters square meters hectares	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47	Newtons Kilopascals FROM SI UNITS To Find inches feet yards miles square inches square feet square yards acres	kPa Symbol in ft yd mi in ² ft ² yd ² ac
Ibl Ibf/in ² Symbol Mm M M m km km ² m ² ha km ²	poundforce per square inch TABLE OF APP When You Know millimeters meters kilometers square millimeters square meters square meters hectares square kilometers	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386	Kewtons Kilopascals FROM SI UNITS To Find inches feet yards miles square inches square feet square yards acres square miles	kPa Symbol in ft yd mi in ² ft ² yd ² ac mi ²
Ibl Ibf/in ² Symbol Mm M M m km km m ² m ² ha km ²	poundforce per square inch TABLE OF APP When You Know millimeters meters meters kilometers square millimeters square meters square meters hectares square kilometers	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME	Kilopascals FROM SI UNITS To Find inches feet yards miles square inches square feet square yards acres square miles	kPa Symbol in ft yd mi in ² ft ² yd ² ac mi ²
Ibl Ibf/in ² Symbol Mm M M m km m ² m ² ha km ² ml	poundforce per square inch TABLE OF APP When You Know millimeters meters kilometers square millimeters square meters square meters hectares square kilometers milliliters	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME 0.034	Kilopascals FROM SI UNITS To Find inches feet yards miles square inches square feet square yards acres square miles	kPa Symbol in ft yd mi in ² ft ² yd ² ac mi ² fl oz
Ibl Ibf/in ² Symbol Mm M M m km m ² m ² ha km ² ha km ² l	poundforce per square inch TABLE OF APP When You Know millimeters meters kilometers square millimeters square meters square meters hectares square kilometers milliliters	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME 0.034 0.264	Kilopascals 5 FROM SI UNITS To Find inches feet yards miles square inches square feet square yards acres square miles fluid ounces gallops	kPa Symbol in ft yd mi in ² ft ² yd ² ac mi ² fl oz gal
Ibl Ibf/in ² Symbol Mm M M m km m ² m ² ha km ² ha km ² mL L m ³	poundforce per square inch TABLE OF APP When You Know millimeters meters kilometers square millimeters square meters square meters hectares square kilometers milliliters itters cubic meters	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME 0.034 0.264 35 314	Kilopascals 5 FROM SI UNITS To Find inches feet yards miles square inches square feet square gards acres square miles fluid ounces gallons cubic feet	kPa Symbol in ft yd mi in ² ft ² yd ² ac mi ² fl oz gal ft ³
Ibl Ibf/in ² Symbol Mm M M m km m ² m ² ha km ² ha km ² mL L m ³ m ³	poundforce per square inch TABLE OF APP When You Know millimeters meters kilometers square millimeters square meters hectares square kilometers milliliters liters cubic meters	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME 0.034 0.264 35.314 1.307	Kilopascals S FROM SI UNITS To Find inches feet yards miles square inches square feet square yards acres square miles fluid ounces gallons cubic feet cubic feet	kPa Symbol in ft yd mi in ² ft ² yd ² ac mi ² fl oz gal ft ³ vd ³
Ibl Ibf/in ² Symbol Mm M M m km m ² m ² ha km ² ha km ² mL L m ³ m ³	poundforce per square inch TABLE OF APP When You Know millimeters meters kilometers square millimeters square meters square meters hectares square kilometers milliliters liters cubic meters cubic meters	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME 0.034 0.264 35.314 1.307 MASS	Kilopascals FROM SI UNITS To Find inches feet yards miles square inches square feet square yards acres square miles fluid ounces gallons cubic feet cubic yards	kPa Symbol in ft yd mi in ² ft ² yd ² ac mi ² fl oz gal ft ³ yd ³
Ibl Ibf/in ² Symbol Mm M M m km m ² m ² ha km ² ha km ² mL L m ³ m ³	poundforce per square inch TABLE OF APP When You Know millimeters meters kilometers square millimeters square meters square meters hectares square kilometers milliliters liters cubic meters cubic meters grams	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME 0.034 0.264 35.314 1.307 MASS 0.035	Newtons Kilopascals FROM SI UNITS To Find inches feet yards miles square inches square feet square yards acres square miles fluid ounces gallons cubic feet cubic yards	kPa Symbol in ft yd mi in ² ft ² yd ² ac mi ² fl oz gal ft ³ yd ³
Ibl Ibf/in ² Symbol Mm M M m km m ² m ² ha km ² mL L m ³ m ³ g	poundforce per square inch TABLE OF APP When You Know millimeters meters kilometers square millimeters square meters square meters hectares square kilometers milliliters liters cubic meters cubic meters grams kilograms	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME 0.034 0.264 35.314 1.307 MASS 0.035 2.202	Kilopascals FROM SI UNITS To Find inches feet yards miles square inches square feet square yards acres square miles fluid ounces gallons cubic feet cubic squares ounces pounds	kPa Symbol in ft yd mi in ² ft ² yd ² ac mi ² fl oz gal ft ³ yd ³ oz lb
Ibl Ibf/in ² Symbol Mm M M m km m ² m ² ha km ² mL L m ³ m ³ g kg	poundforce per square inch TABLE OF APP When You Know millimeters meters kilometers square millimeters square meters square meters hectares square kilometers milliliters liters cubic meters cubic meters grams kilograms megagrams (or "metric tor")	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME 0.034 0.264 35.314 1.307 MASS 0.035 2.202 1.102	Newtons Kilopascals FROM SI UNITS To Find inches feet yards miles square inches square feet square yards acres square miles fluid ounces gallons cubic feet cubic yards ounces pounds short tops (2000 lb)	kPa Symbol in ft yd mi in ² ft ² yd ² ac mi ² fl oz gal ft ³ yd ³ Oz lb T
Ibl Ibf/in ² Symbol Mm M m km m ² m ² ha km ² mL L m ³ m ³ g kg Mg (or "t")	poundforce per square inch TABLE OF APP When You Know millimeters meters meters kilometers square millimeters square meters square meters hectares square kilometers milliliters liters cubic meters cubic meters grams kilograms megagrams (or "metric ton")	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME 0.034 0.264 35.314 1.307 MASS 0.035 2.202 1.103 TEMDED ATUDE	Kilopascals FROM SI UNITS To Find inches feet yards miles square inches square feet square yards acres square miles fluid ounces gallons cubic feet cubic yards ounces pounds short tons (2000 lb) Tomestum in in surrat deserver	kPa Symbol in ft yd mi in ² ft ² yd ² ac mi ² fl oz gal ft ³ yd ³ oz lb T
Ibl Ibf/in ² Symbol Mm M M m km m ² m ² ha km ² mL L m ³ m ³ g kg Mg (or "t")	poundforce per square inch TABLE OF APP When You Know millimeters meters meters kilometers square millimeters square meters square meters hectares square kilometers milliliters liters cubic meters cubic meters grams kilograms megagrams (or "metric ton")	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME 0.034 0.264 35.314 1.307 MASS 0.035 2.202 1.103 TEMPERATURE 4.92	Newtons Kilopascals FROM SI UNITS To Find inches feet yards miles square inches square feet square yards acres square miles fluid ounces gallons cubic feet cubic yards ounces pounds short tons (2000 lb) Temperature is in exact degrees	kPa Symbol in ft yd mi in ² ft ² yd ² ac mi ² fl oz gal ft ³ yd ³ oz lb T
Ibl Ibf/in ² Symbol Mm M M m km m ² m ² ha km ² mL L m ³ m ³ g kg Mg (or "t")	poundforce per square inch TABLE OF APP When You Know millimeters meters meters kilometers square millimeters square meters square meters hectares square kilometers milliliters liters cubic meters cubic meters grams kilograms megagrams (or "metric ton")	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME 0.034 0.264 35.314 1.307 MASS 0.035 2.202 1.103 TEMPERATURE 1.8c + 32	Kilopascals FROM SI UNITS To Find inches feet yards miles square inches square feet square yards acres square miles fluid ounces gallons cubic feet cubic yards ounces pounds short tons (2000 lb) Temperature is in exact degrees Fahrenheit	kPa Symbol in ft yd mi in ² ft ² yd ² ac mi ² fl oz gal ft ³ yd ³ oz lb T °F
Ibl Ibf/in ² Symbol Mm M m km m ² m ² ha km ² mL L m ³ m ³ g kg Mg (or "t") °C	poundforce per square inch TABLE OF APP When You Know millimeters meters meters kilometers square millimeters square meters square meters square meters square meters square kilometers milliliters liters cubic meters cubic meters megagrams (or "metric ton") Celsius	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME 0.034 0.264 35.314 1.307 MASS 0.035 2.202 1.103 TEMPERATURE 1.8c + 32 ILLUMINATION	Newtons Kilopascals FROM SI UNITS To Find inches feet yards miles square inches square feet square yards acres square miles fluid ounces gallons cubic feet cubic yards ounces pounds short tons (2000 lb) Temperature is in exact degrees Fahrenheit	kPa Symbol in ft yd mi in ² ft ² yd ² ac mi ² fl oz gal ft ³ yd ³ oz lb T °F
Ibl Ibf/in ² Symbol Mm M M m km m ² m ² ha km ² mL L m ³ m ³ g kg Mg (or "t") °C Ix	poundforce per square inch TABLE OF APP When You Know millimeters meters meters kilometers square millimeters square meters square meters hectares square kilometers milliliters liters cubic meters cubic meters grams kilograms megagrams (or "metric ton") Celsius	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME 0.034 0.264 35.314 1.307 MASS 0.035 2.202 1.103 TEMPERATURE 1.8c + 32 ILLUMINATION 0.0929	Newtons Kilopascals FROM SI UNITS To Find inches feet yards miles square inches square feet square yards acres square miles fluid ounces gallons cubic feet cubic yards ounces pounds short tons (2000 lb) Temperature is in exact degrees Fahrenheit	kPa Symbol in ft yd mi in ² ft ² yd ² ac mi ² fl oz gal ft ³ yd ³ oz lb T °F fc
Ibl Ibf/in ² Symbol Mm M M m km m ² m ² ha km ² mL L m ³ m ³ g kg Mg (or "t") °C Ix cd/m ²	poundforce per square inch TABLE OF APP When You Know millimeters meters meters kilometers square millimeters square meters square meters hectares square kilometers milliliters liters cubic meters cubic meters grams kilograms megagrams (or "metric ton") Celsius lux candela/m ²	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME 0.034 0.264 35.314 1.307 MASS 0.035 2.202 1.103 TEMPERATURE 1.8c + 32 ILLUMINATION 0.0929 0.2919	Kilopascals FROM SI UNITS To Find inches feet yards miles square inches square feet square yards acres square miles fluid ounces gallons cubic feet cubic feet cubic short tons (2000 lb) Temperature is in exact degrees Fahrenheit foot-candles foot-Lamberts	kPa Symbol in ft yd mi in ² ft ² yd ² ac mi ² fl oz gal ft ³ yd ³ oz lb T °F fc fl
Ibl Ibf/in ² Symbol Mm M M m km m ² m ² ha km ² mL L m ³ m ³ g kg (or "t") °C Ix cd/m ²	poundforce per square inch TABLE OF APP When You Know millimeters meters meters kilometers square millimeters square meters square meters hectares square kilometers milliliters liters cubic meters cubic meters grams kilograms megagrams (or "metric ton") Celsius lux candela/m ²	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME 0.034 0.264 35.314 1.307 MASS 0.035 2.202 1.103 TEMPERATURE 1.8c + 32 ILLUMINATION 0.0929 0.2919 Force & Pressure Or Stress	Newtons Kilopascals FROM SI UNITS To Find inches feet yards miles square inches square feet square yards acres square miles fluid ounces gallons cubic feet cubic yards ounces pounds short tons (2000 lb) Temperature is in exact degrees Fahrenheit foot-candles foot-Lamberts	kPa Symbol in ft yd mi in ² ft ² yd ² ac mi ² fl oz gal ft ³ yd ³ oz lb T °F fc fl
Ibl Ibf/in ² Symbol Mm M M m km m ² m ² ha km ² mL L m ³ m ³ g kg Mg (or "t") °C Ix cd/m ² N	poundforce per square inch TABLE OF APP When You Know millimeters meters meters kilometers square millimeters square meters square meters hectares square kilometers milliliters liters cubic meters cubic meters grams kilograms megagrams (or "metric ton") Celsius lux candela/m ² newtons	4.45 6.89 ROXIMATE CONVERSIONS Multiply By LENGTH 0.039 3.28 1.09 0.621 AREA 0.0016 10.764 1.195 2.47 0.386 VOLUME 0.034 0.264 35.314 1.307 MASS 0.035 2.202 1.103 TEMPERATURE 1.8c + 32 ILLUMINATION 0.0929 0.2919 Force & Pressure Or Stress 0.225	Newtons Kilopascals FROM SI UNITS To Find inches feet yards miles square inches square feet square yards acres square miles fluid ounces gallons cubic feet cubic yards ounces pounds short tons (2000 lb) Temperature is in exact degrees Fahrenheit foot-candles foot-Lamberts poundforce	kPa Symbol in ft yd mi in ² ft ² yd ² ac mi ² fl oz gal ft ³ yd ³ oz lb T °F fc fl lbf

* SI is the symbol for the International System of Units. Appropriate rounding should be made to comply with Section 4 of ASTM E380. (Revised March 2003, Section 508-accessible version September 2009)

TABLE OF CONTENTS

LIS	T OF	ACRONYMS, ABBREVIATIONS, AND SYMBOLS	•X
EXE	ECUT	IVE SUMMARYx	iii
1.	BAC	KGROUND	.1
2.	EXA	MINATION OF PINHOLE LEAKS	3
	2.1	BACKGROUND: POSSIBLITY OF CORROSION MECHANISMS FOR FORMING PINHOLE DEFECTS	3
	2.2	BACKGROUND: WELDING DEFECTS THAT CAN FORM PINHOLE DEFECT	'S5
	2.3	FMCSA EXAMINATION OF MANUFACTURER'S WELDING PROCEDURES.	8
	2.4	OUTCOMES OF THE TASKS PERFORMED TO EXAMINE PINHOLE LEAKS IN NURSE TANKS	9
		2.4.1 Acquired five nurse tanks that contained pinhole leaks	9
		2.4.2 Performed visual and angle-beam ultrasound testing of donated tanks	9
		2.4.3 Cut into the tanks and removed coupons for metallographic study	11
		2.4.4 Performed wet magnetic particle testing of the inner tank walls	11
		2.4.5 Performed serial machining on pinhole coupon.	12
		2.4.6 Transported a pinhole leak coupon section to LANSCE	13
		2.4.7 Assessed findings to determine the cause(s) of the pinhole leaks	13
		2.4.8 Radiographic inspection of a pinhole leak	14
	2.5	CONCLUSIONS ON CAUSE OF PINHOLE LEAKS	17
		2.5.1 Probable Cause of Pinhole Leaks	17
		2.5.2 Low Likelihood of Catastrophic Failure of the Tank	17
3.	STR	ESS CORROSION CRACK GROWTH MEASUREMENT	19
	3.1	THE SCC EXPERIMENTAL PLAN	19
	3.2	WET MAGNETIC PARTICLE INSPECTION OF TEST TANKS	24
	3.3	CONCLUSIONS FROM SCC TESTING	27
4.	RES	IDUAL STRESS MEASUREMENTS IN AN ANNEALED NURSE TANK	28
	4.1	RESIDUAL STRESS IN WELDMENTS	29
	4.2	RESIDUAL STRESS MEASUREMENTS OF UNANNEALED TANKS	31
	4.3	IMPLICATIONS OF THE RESIDUAL STRESS FINDINGS FOR SCC IN NURSE TANKS] 36
	4.4	RESIDUAL STRESS MEASUREMENTS OF ANNEALED TANKS	37
	4.5	FINDINGS FROM NEUTRON DIFFRACTION WORK	38

	4.6	CONCLUSIONS FROM RESIDUAL STRESS MEASUREMENT IN AN ANNEALED NURSE TANK	41
5.	SUF	RVEYOF DEFECTS IN NURSE TANKS	43
	5.1	INTRODUCTION	43
	5.2	METHODOLOGY	43
	5.3	RESULTS	53
		5.3.1 Effect of Stress Relief	56
	5.4	POSSIBLE DIFFERENCES IN FABRICATION PROCEDURES	63
	5.5	EXAMINATION OF LEG WELDS	66
	5.6	OVERALL SUMMARY OF THE INDICATIONS	68
	5.7	DISCUSSION	69
		5.7.1 Number and Types of Indications	69
		5.7.2 Effects of Tank Age, Size, and Thickness	75
	5.8	SUMMARY AND CONCLUSIONS	77
6.	SUN FUI	IMARY OF KEY FINDINGS FROM THE PROJECT AND POSSIBLE TURE RESEARCH	79
	6.1	WHAT HAS BEEN LEARNED?	
	6.2	WHAT ACTIONS ARE RECOMMENDED BASED UPON THESE FINDING	3S?.80
	6.3	WHAT REMAINS UNKNOWN THAT WOULD BE VALUABLE IF KNOW	N?82
		6.3.1 Ultrasound Inspection	82
		6.3.2 Corrosion Inhibitors/Corrosion Barriers	82
		6.3.4 Effects of Steel Composition on Stress Corrosion Cracking	82 82
		6.3.5 Acoustic Emission	02
		6.3.6 Number of Indications Per Tank	83
	6.4	HOW COULD THE UNKNOWNS BE DETERMINED BY FUTURE RESEA	RCH?83
		6.4.1 Ultrasound Inspection	83
		6.4.2 Corrosion Inhibitors/Corrosion Barriers	84
		6.4.3 Post-weld Heat Treatment	84
		6.4.4 Effects of Steel Composition on Stress-corrosion Cracking	84
		6.4.5 Effects of Steel Composition and Thickness on Stress Corrosion Crackin	1g 84
		6.4.6 Acoustic Emission	84
REI	FERE	NCES	99

LIST OF APPENDICES

LIST OF FIGURES (AND FORMULAS)

Figure 1. Diagram. Typical damage geometries for various types of corrosion in metal plates4
Figure 2. Image. Cold lap/incomplete fusion depicted in a radiograph
Figure 3. Illustration. Cold lap/incomplete fusion depicted in a sketch
Figure 4. Image. X-ray radiography image of cracks in a weld
Figure 5. Illustration. Weld deposit cracks depicted in a sketch
Figure 6. Image. X-ray radiography image of bubble porosity in a weld7
Figure 7. Illustration. Bubble porosity in a weld, depicted in a sketch
Figure 8. Photograph. Exterior of a pinhole located at the vapor valve (same pinhole is depicted
in Figure 9)
Figure 9. Grouped photograph. Metallographic sections of a serially-machined pinhole leak
defect
Figure 10. Photograph. Two pinhole leaks in the roundseam weld of 1,450-gallon tank
9NU00188215
Figure 11. Photograph. Radiographic image of section of center girth weld showing radio-opaque
markers (1 and 2) placed at the locations of the pinholes
Figure 12. Photograph. Close-up of the weld shown in Figure 11.
Figure 13. Rendering. Design for constant-load SENT SCC samples
Figure 14. Scatterplot. Crack length after 3 months of exposure
Figure 15. Scatterplot. Crack length after 6 months of exposure
Figure 16. Equation. Lunde and Nyborg crack length in meters after y years
Figure 17. Equation. Phase I crack length in meters after y years
Figure 18. Equation. Phase II crack length in meters after <i>y</i> years22
Figure 19. Photograph. Sample #055 before the sample was inserted into the tank
Figure 20. Photograph. Sample #055 after 3 months of exposure to liquid ammonia23
Figure 21. Photograph. Sample #055 after 15 months of exposure to liquid ammonia23
Figure 22. Photograph. Magnetic particle inspection of a weld region, before 12 months of
exposure to NH_3
Figure 23. Photograph. Magnetic particle inspection of the same weld region shown in Figure 22, after 12 months of exposure to NH ₃
Figure 24. Photograph. The rod from the fill gauge had corroded off its fitting in the tank containing N-Serve
Figure 25. Photograph. The float gauges from the other tanks (not containing N-Serve) were still whole
Figure 26. Image. Wave form indicates normal status, neither compressed or stretched
Figure 27. Image. Wave form indicates compressed status
Figure 28. Image. Wave form indicates stretched status
Figure 29. Diagram. Hoop weld sections (12 inches wide) were cut from tanks and examined by neutron diffraction at LANSCE.
Figure 30. Photograph. Nurse tank hoop section in the neutron beam line at LANSCE

Figure 31. Scatterplot. Residual hoop stress distributions measured by neutron diffraction at LANSCE; tensile stresses are positive, while compressive stresses are negative	34
Figure 32. Line graph. Superimposition of the maximum residual tensile stress measured at LANSCE (lower horizontal line) with the stress-strain plot for the steel coupons measured from that tank in tensile testing	35
Figure 33. Graph. Furnace temperature profile for 1.000-gallon nurse tank (serial #6NF001939	€).37
Figure 34. Graph. Hoop stress across circumferential weld in a fully strain-relieved (S.R.) nurs	sé
tank manufactured in 2011 (solid lines) overlaid with an unannealed tank manufactu in 1966 (dashed lines).	red 39
Figure 35. Graph. Axial stress across circumferential weld in a fully S.R. nurse tank	
manufactured in 2011	40
Figure 36. Graph. Radial stress across circumferential weld in a fully S.R. nurse tank manufactured in 2011.	40
Figure 37. Diagram. Schematic of a transducer and a 45-degree wedge	45
Figure 38. Photograph. Student conducting an examination of a nurse tank	47
Figure 39. Photograph. Close-up of inspection of a nurse tank	47
Figure 40. Photograph. Close-up of the unit used to conduct nurse tank inspections	47
Figure 41. Screenshot. First page of nurse tank inspection form	48
Figure 42. Screenshot. Second page of nurse tank inspection form.	49
Figure 43. Screenshot. Third page of nurse tank inspection form.	50
Figure 44. Screenshot. Fourth page of nurse tank inspection form.	51
Figure 45. Screenshot. Fifth page of nurse tank inspection form.	52
Figure 46. Bar graph. Summary of tank manufacturers and the number of tanks examined	53
Figure 47. Scatterplot. Tanks inspected as a function of year of manufacture	54
Figure 48. Scatterplot. Number of indications as a function of year of manufacture	55
Figure 49. Graph. Number of indications per tank for strain-relieved (S.R.) tanks	57
Figure 50. Graph. Alternate representation of data from Figure 49.	58
Figure 51. Scatterplot. Bivariate fit of indication number (per tank) by age of tank in years	59
Figure 52. Scatterplot. Bivariate fit of indication number (per tank) by age of tank in years for tanks manufactured from 1999-2011.	60
Figure 53. Graph. Indications per tank as a function of tank size.	61
Figure 54. Graph. Indications per tank by head shape	62
Figure 55. Graph. Comparison of number of indications for inspected tanks manufactured afte 1998.	r 65
Figure 56. Graph. Number of indications for inspected tanks manufactured after 1998	66
Figure 57. Scatterplot. Nurse tanks with indications at leg welds as a percentage of total tanks	
tested	67
Figure 58. Scatterplot. Nurse tanks with indications at leg welds as an absolute number of tank	cs.67
Figure 59. Photograph. Example of one type of tank handle	70
Figure 60. Diagram. Schematic showing how a third 'leg' response in the acoustic signal can be caused by a gap between the lug and the base metal.	эе 70

Figure 61. Diagram. Figure UW-16.1 (1) adapted from Section VIII, Div. 1 of ASME 2011a Section 1 of the Boiler and Pressure Vessel Code
Figure 62. Diagram. Schematic with a cross-section of a lap joint showing why a signal is generated at a weld containing a blind corner
Figure 63. Diagram. A cross-section of a lap joint showing full penetration of the weld bead72
Figure 64. Graph. Tanks with critical crack lengths, assuming through-cracking exists,
Figure 65. Bar graph. Location of HAZ indications in a population of 532 nurse tanks
Figure 66. Grouped photograph. Magnetic particle highlighted examples of crack branching in nurse tank SCCs
Figure 67. Flowchart, Revised recommendation for nurse tank inspection procedures
Figure 68. Photograph. Illegible data plate
Figure 69. Photograph. Pinhole leak on liquid vapor valve on nurse tank DOT SP 13554
Figure 70. Photograph. Close-up of pinhole leak on the liquid vapor valve of nurse tank DOT SP 13554
Figure 71. Photograph. Same pinhole leak after cleaning the site with Calcium Lime Rust (CLR) remover
Figure 72. Photograph. Reverse side of same pinhole showing underside of weld
Figure 73. Photograph. Serial mill picture of pinhole path
Figure 74. Photograph. Data plate for nurse tank 6NU001139, which was certified in 2010 by AWT
Figure 75. Photograph. Pinhole leak on vapor valve on nurse tank 6NU001139
Figure 76. Photograph. Close-up of pinhole leak on vapor valve on nurse tank 6NU00113990
Figure 77. Photograph. Close-up of pinhole leak on vapor valve on nurse tank 6NU001139 after being cleaned with CLR
Figure 78. Photograph. The data plate for nurse tank 9NF000057, which was certified by AWT in 2005
Figure 79. Grouped photograph. Location of leak on leg weld of nurse tank 9NU00005791
Figure 80. Photograph. The crack from inside the tank under ultraviolet illumination
Figure 81. Photograph. A cross-section of the tank shell at the end of the leg weld
Figure 82. Photograph. Opened-up weld side of crack in tank shell
Figure 83. Photograph. Opened-up crack in tank shell, anti-crack side
Figure 84. Photograph.Displays how river lines begin as closely-spaced steps which merge into larger and more widely-spaced steps as the fracture progresses
Figure 85. Photograph. Data plate for nurse tank 9NU001882, which was certified by AWT in 2007
Figure 86. Photograph. Nurse tank 9NU001882 had two pinholes on the center circumferential weld
Figure 87. Image. An 18-inch flaw found on the shell side of the interior of a head-shell circumferential weld
Figure 88. Photograph. Nurse tank #655 had a pinhole leak along one of the weld seams
Figure 89. Photograph. Nurse tank #655 did not have a data plate
Figure 90. Photograph. This tank had a pinhole leak where the head was welded to the tank shell.98

LIST OF TABLES

Table 1. Tanks donated for pinhole leak study.	9
Table 2. Visual inspection of flaws	10
Table 3. Angle-beam ultrasound inspection results.	10
Table 4. Fluorescent wet magnetic particle inspection.	11
Table 5. Maximum hoop, axial, and radial stresses in a nurse tank resulting from internalpressure and from residual stress in the HAZ near welds.	36
Table 6. Number of indications in tanks with or without full strain relief	57
Table 7. Mean and standard deviation data on the number of indications for populations of tank with elliptical and hemispherical heads.	cs 62
Table 8. Mean and standard deviation for data shown in Figure 55	65
Table 9. Number of indications, mean, and standard deviation data on the number of indication for 1,450-gallon, 1,000-gallon, and stress-relieved tanks surveyed.	s 76
Table 10. Number of indications, mean, and standard deviation data for tanks manufactured before 1999.	76

LIST OF ACRONYMS, ABBREVIATIONS, AND SYMBOLS

Acronym	Definition
ANSI	American National Standards Institute
ASME	American Society of Mechanical Engineers
ASTM	formerly known as the American Society for Testing and Materials
AWS	American Welding Society
AWT	American Welding & Tank
C	Celsius
CLR®	Calcium, Lime, Rust Remover
CNDE	Center for Nondestructive Evaluation
F	Fahrenheit
FMCSA	Federal Motor Carrier Safety Administration
HAZ	heat-affected zone
HMSP	Hazardous Materials Safety Permit
IDALS	Iowa Department of Agriculture and Land Stewardship
ISU	Iowa State University
ksi	kilopounds per square inch
LANSCE	Los Alamos Neutron Science Center
MPa	MegaPascal
$MPa(m)^{0.5}$	MegaPascal square root meter
NBBI	National Board of Boiler and Pressure Vessel Inspectors
NDE	non-destructive examination
NTSB	National Transportation Safety Board
psi	pounds per square inch
PWHT	post-weld heat treatment
R ²	coefficient of determination

Acronym	Definition
SCC	stress corrosion crack
SENT	single-edge notched tension
SMARTS	Spectrometer for Materials Research at Temperature and Stress
S.R.	Strain-relieved (i.e., post-weld heat-treated to lower residual stress)

CHEMICAL ABBREVIATIONS

Al	Aluminum
Br ⁻¹	Bromide ion
Cl ⁻¹	Chloride ion
ClO ⁻¹	Hypochlorite ion
Cr	Chromium
Fe(OH) _x	Rust, iron hydroxide
Н	Hydrogen
H ₂ O	Water
Мо	Molybdenum
Mn	Manganese
N_2	Nitrogen
NH ₃	Anhydrous Ammonia
Ni	Nickel
O ₂	Oxygen
Р	Phosphorus
S	Sulfur
Si	Silicon
Тс	Technetium
Ti	Titanium
Zn	Zinc
Zr	Zirconium

EXECUTIVE SUMMARY

PURPOSE

This report presents findings of four additional research activities performed as Phase II of nurse tank research at Iowa State University (ISU). Phase II was initiated in October 2010 to study stress corrosion cracks (SCCs), pinhole leaks, and related phenomena in nurse tanks used to transport anhydrous ammonia (NH₃). Phase I was initiated in December 2008 and a final report for that phase was issued on October 2013. NH₃, which is typically used in the agricultural field as a nitrogen-rich fertilizer, is a hazardous material that can cause chemical burns, frostbite, and suffocation. Steel nurse tanks hold NH₃ at multiple times atmospheric pressure. Tank failures can release this pressure with explosive force, posing the additional risk of impact injury to workers and bystanders. The four follow-on activities addressed by Phase II are as follows:

- 1. **Pinhole leaks**: This study concluded that the principal cause of pinhole leaks next to welds is porosity in welds that subsequently interconnects from stresses in operations. Such porosity usually results from welding steel with surface contamination from water, mill scale, rust, and other contaminants. Such pinhole leaks can be avoided by following the welding procedure laid out here: American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code, VIII, Div. 1, UW-30, UW-31, & UW-32.
- 2. **Stress-relief annealing**: Residual tensile stress is a key factor in initiating and enabling SCCs to grow in nurse tanks. SCCs are a primary cause of tank failures, which lead to injuries and fatalities. Residual stress occurs from the thermal stress induced by welding the tanks during manufacture. It has long been known that stress-relief annealing (heating the welded tank in a furnace) assists the atomic structure of the stressed steel to relax some, thus lowering residual tensile stress in such heat-treated (annealed) steel. In this study, the headto-shell stress in a tank with ASME protocol stress-relief annealing was measured by neutron diffraction analysis to determine the residual tensile stress levels. These measurements were compared to previous measurements made on two unannealed tanks, and it was found that the ASME-recommended protocol for stress-relief annealing lowered the residual tensile hoop stress (the largest stress in the tanks) by two-thirds. It lowered the smaller axial residual tensile stress by one-third. These large stress reductions put the remaining residual stresses well within the elastic range of the steel and thus are predicted to greatly extend tank lifetimes and lower risks related to stress corrosion cracking. The field testing (described below) for cracks in unannealed and annealed nurse tanks significantly supports this. Namely, almost no significant indications of possible cracks were found in 104 stress-relieved tanks.
- 3. **Nurse tank crack survey**: A side-angle ultrasound survey of 532 in-service nurse tanks was performed to measure ultrasound indications, most of which were indications of cracks in the tanks' steel walls. This survey showed that many tanks contain indications, but nearly all of these are too short and too shallow to pose an immediate risk of tank failure. The 104 tanks that had received stress-relief annealing had dramatically reduced numbers and size of indications. The majority of ultrasound indications were found in the unannealed heat-affected zone (HAZ) near welds (i.e., the metal that got hot during welding but did not

actually melt). Most indications lay perpendicular to head-to-shell welds. Likewise, most of the indications occurred at or above the 80-percent fill line. In this portion of the tank, ammonia vapor (with no water) contacts the tank's inner wall surface. Below the 80-percent fill line, liquid ammonia with water contacts the tank's inner wall surface.

4. **Stress corrosion cracking study**: This study exposed 200 steel specimens for 12–15 months to various environments while holding the steel under high tensile loads for the entire time period. It was expected that the test would cause some specimens to initiate and grow SCCs. Three different environments were tested: ammonia + 0.2 percent water, ammonia + 0.2 percent water + N-Serve (an additive that improves the fixing of nitrogen to soil particles in environmental conditions when it would otherwise be rapidly lost); and ammonia + 0.2 percent water with the tank purged with pure nitrogen gas each time before the tank was loaded. In each test, some specimens were immersed in liquid ammonia, while other specimens were exposed to the vapor in the tank space above the liquid. Specimens were removed from the ammonia and examined periodically to note crack formation and growth rates, then returned to the test environment for further exposure. The amount of crack initiation and growth in Phase II was less than anticipated in all six environments from what was observed in Phase I. The data obtained from this portion of the study (for both Phase I and Phase II) are not adequate to allow confident predictions of crack growth rates in actual tank service. The study did show that tanks containing N-Serve experience higher uniform corrosion rates and should be monitored for minimum wall thickness. Additional study of SCC propagation rates in ammonia would be desirable. This could be accomplished by periodically replicating the field measurements of the indications discovered on the 532 tanks.

The information collected during this project has aided in the development of recommendations for best inspection practices to reduce risks associated with nurse tank failures.

This report may interest nurse tank owners, manufacturers, repair businesses, and farmers using nurse tanks to fertilize their crops, as well as all other parties concerned about public roadway safety. This document is the second report resulting from this ongoing research.

1. BACKGROUND

This is the second report in a two-phased, multi-year research project examining options for, and practicality of, non-destructive examination (NDE) of nurse tanks used to transport anhydrous ammonia (NH₃). NH₃, often used as a nitrogen fertilizer, is a hazardous material that can cause chemical burns, frostbite, and suffocation. Steel nurse tanks hold NH₃ at multiple times atmospheric pressure. Tank failures can release this pressure with catastrophic force, posing the additional risk of impact injury to workers and bystanders.

This report focuses on measures of practical metrics. As such, it extends and builds on the theoretical results presented in the first report. Based on recommendations received from the advisory group assisting with this research, the work accomplished under Phase I was expanded into Phase II in order to address the following major issues related to nurse tank safety:

- Determining the safety of nurse tanks discovered to have pinhole leaks. The issue was whether pinhole leaks in a nurse tank indicate a structural weakness, and thus a risk of catastrophic failure. To address this, researchers examined the structure, causes, and possible remediation actions for pinhole leaks that have been discovered in nurse tanks. These pinhole leaks tend to be narrow apertures in welds (less than 1mm in diameter) that vent NH₃ into the atmosphere relatively slowly. However, the possibility of sudden, catastrophic rupture was a concern.
- Gathering more definitive information on the rate of crack growth by performing stress tests over a more extended period of time. Researchers performed tests to measure stress corrosion crack (SCC) growth rates in steel specimens loaded in tension and exposed to NH₃ in three test tank environments. One tank contained NH₃, a second tank contained NH₃ with N-Serve added, and a third tank contained NH₃ with a nitrogen purge to ensure there was not oxygen in the tank. Crack growth data can guide failure predictions and inspection methods.
- Determining the effectiveness of annealing (a post-weld heat treatment [PWHT] performed to reduce residual stress in the metal) in reducing residual stresses. Researchers compared residual stresses in annealed and unannealed nurse tanks. Annealing a tank after it is joined by welding can lower the residual stresses in the metal near welds. Lower tensile residual stresses reduce the driving force for SCC initiation in the tank steel. Neutron diffraction was used to determine the stress state of regions in and adjacent to welds. The original Phase I research examined two unannealed tanks. This Phase II follow-on research looked at a tank annealed using the American Society of Mechanical Engineers (ASME)-recommended protocol for annealing.
- Determining the distribution of SCCs by tank type, and by location in the tanks. Researchers surveyed 532 in-service nurse tanks of varying ages and manufacturers using side-angle ultrasound inspection. They cataloged the strength, orientation, and numbers of ultrasound indications associated with cracks in the tank wall. Such a large-scale survey had not been undertaken previously, and the results of the survey provide an overview of the existing flaw population in a somewhat representative sample of the U.S.

nurse tank fleet located in Iowa, within commuting distance of Iowa State University (ISU).

This Phase II final report presents findings from the above-outlined research activities completed by a research team at ISU. This material draws on findings from the Phase I research performed from December 2008 to May 2011 by ISU.

The information collected during this project serves as a guide for developing recommendations for best inspection practices to reduce risks associated with nurse tank failures.

2. EXAMINATION OF PINHOLE LEAKS

The following tasks were completed when performing examination of pinhole leaks in nurse tanks:

- Acquired five nurse tanks that contained pinhole leaks.
- Performed visual and side-angle ultrasound testing on these tanks.
- Cut into tanks and removed coupons (samples) for metallographic study to determine the cause of the leaks. Performed wet magnetic particle testing of the inner tank walls to detect cracks.
- Performed serial machining to measure the internal contours of a pinhole cross-section as it penetrated through the steel wall.
- Cut a test coupon from the metal around a pinhole leak for residual stress analysis at Los Alamos Neutron Science Center (LANSCE).
- Transported the pinhole leak coupon section to LANSCE. Unfortunately, researchers were unable to measure the residual stress in that sample due to reduction in available beam time. (Beam time was reduced for all users who were granted time and denied entirely to other users after a radioactive material spill at LANSCE in August, 2012. This project's reduced beam time was used to measure residual stresses in a post-weld heat-treated nurse tank, because this was deemed the most important measurement for this phase's study. The next LANSCE beam cycle would not begin until after this project's extended end date. Thus, researchers:
 - Performed radiography on a weld section containing pinhole leaks.
 - Made a determination of the cause(s) of the pinhole leaks studied.

In 2009, the Federal Motor Carrier Safety Administration (FMCSA) began an investigation of a nurse tank manufacturer whose supplied tanks (which were very new) were developing pinhole leaks in circumferential welds and in coupling attachment welds. NH₃ leaked slowly from these tanks due to the small size of pinhole defects. These tanks were removed from service because the leak rates of ammonia were high enough that the ammonia posed a hazard to workers, and there was concern that a catastrophic ammonia release could occur.

2.1 BACKGROUND: POSSIBLITY OF CORROSION MECHANISMS FOR FORMING PINHOLE DEFECTS

Most corrosion damage occurs in metal parts by one of three means (see Figure 1):

- Uniform loss of metal at a surface.
- Attack in a thin, crack-shaped geometry.
- Loss of metal in irregularly-shaped depressions.

All of these geometries are inconsistent with the long, narrow, irregular holes observed in nurse tanks containing pinholes, which suggests that the pinhole flaws were probably not caused by any of these means of corrosive attack.

However, a less-common form of corrosion can produce damage that might be more similar to the geometry of a pinhole defect. Pitting corrosion is an extremely localized attack that produces holes in metal. These holes vary considerably in shape and diameter, but they are typically a few millimeters in diameter. Pits tend to move downward on the bottom of tanks—not upward—through the metal. This is because they are most commonly initiated by oxygen-depleted regions in stagnant fluid trapped under debris at the bottom of a tank. This can produce a low-pH (acidic) solution in the small volume of liquid lying between the debris and the metal, which accelerates the corrosion rate. The debris frequently sinks slowly into the pit as corrosion progresses, maintaining the oxygen-depleted, low-pH condition as it moves.

For this to occur, the tank most likely needs to sit idle so that it does not experience liquid refresh from refilling and emptying and so that it is not being towed around on rough terrain, which causes sloshing of the ammonia. Time is required for the pitting corrosion to progress. These requirements (idle tanks that are sitting over time) do not seem to apply to newly-manufactured nurse tanks that are developing pinhole leaks.

In some cases, pitting corrosion can initiate by random fluctuations in ion concentrations on a clean surface. Stagnation of the fluid within the pit often causes progressively larger shifts in solution pH and/or anode-to-cathode area ratios make the attack autocatalytic (i.e., the deeper the pit becomes, the faster it advances into the metal).



Figure 1. Diagram. Typical damage geometries for various types of corrosion in metal plates.

Pitting normally requires an initiation period that ranges from months to years. Once a pit begins to grow, it can deepen at an increasing pace, often undercutting the surface to produce a roughly conical channel in the metal, narrow at the initiation site but widening as it moves deeper into the metal (Figure 1). Pitting is an insidious form of corrosion in pressure vessels because initiation usually occurs on the tank's inner surface, and there is no externally visible damage until perforation occurs, causing a sudden leak.

For all the reasons given, even pitting corrosion does not look like it is the mechanism by which the observed pinholes in newly-manufactured nurse tanks were formed.

2.2 BACKGROUND: WELDING DEFECTS THAT CAN FORM PINHOLE DEFECTS

Crack-like discontinuities in welds can occur due to various causes:

• Cold lap or incomplete fusion occurs when the weld filler metal fails to locally fuse with the base metal. The arc does not melt the base metal sufficiently, causing the molten puddle to flow onto the base material without bonding to it. In an ideal weld, both the base metal and the filler metal will be molten so that they can co-mingle and form the strongest possible joining of the metals. Incomplete fusion can cause leaks in pressure vessels when the NH₃ penetrates the entire weld, leaving a narrow gap that allows ammonia to pass through the weld. Incomplete fusion is often difficult to see on a radiograph, as shown in Figure 2. When visible, it appears as a dark line or region oriented in the direction of the weld seam and positioned to one side of the weld centerline. Examples of incomplete fusion are illustrated more clearly in the sketch shown in Figure 3.



Figure 2. Image. Cold lap/incomplete fusion depicted in a radiograph.

Source: The Center for Nondestructive Evaluation (CNDE), ISU.



Figure 3. Illustration. Cold lap/incomplete fusion depicted in a sketch. Source: The CNDE, ISU.

Weld cracks are more problematic in high-hardenability steels (e.g., alloy steels containing carbon-diffusion retarding agents such as chromium (Cr), nickel (Ni), or molybdenum (Mo) or in medium-carbon or high-carbon steels). In these steels, rapid cooling of the weld joint can form brittle martensite, which sometimes cracks when subjected to the thermally-induced stresses that occur as the weld cools to room temperature. However, steels used to fabricate nurse tanks are not high-hardenability steels. Steels used in nurse tanks are low-carbon steels with minimal alloying additions. Weld deposit cracks can be detected in a radiograph (see Figure 4) when they are propagating in a direction parallel to the x-ray beam. Examples of weld deposit cracks are also shown in the sketch portrayed in Figure 5. Cracks will appear as jagged and often very faint irregular lines, sometimes as "tails" on inclusions or porosity.



Figure 4. Image. X-ray radiography image of cracks in a weld. Source: The CNDE, ISU.



Figure 5. Illustration. Weld deposit cracks depicted in a sketch.

Source: The CNDE, ISU.

Porosity is a defect sometimes seen in metals joined by welding. Porosity often appears as bubbles or voids inside the weld zone (see Figure 6 and Figure 7). One cause of weld porosity is inadequate preparation of fraying surfaces.⁽¹⁾ Fraying surfaces are materials that are in contact

with each other and that are joined or about to be joined together. In the case of nurse tanks, the fraying surfaces are joined by welding. Water, oil, paint, mill scale, dirt, or rust residues on the metal surface can be vaporized by the heat of the welding operation.



Figure 6. Image. X-ray radiography image of bubble porosity in a weld. Source: The CNDE, ISU.



Figure 7. Illustration. Bubble porosity in a weld, depicted in a sketch.

Source: The CNDE, ISU.

Some vapors can dissolve into the molten metal, but they cannot continue to be held by it when it cools and returns to a solid state. The vapors are rejected at the advancing liquid/solid metal interface, and there they form bubbles or a series of bubbles. Such porosity occurs when gas being released by the freezing metal attempts to escape to the surface along the solid-liquid interface but does not reach the surface before the metal freezes. Such a series of bubbles is what appears to have occurred. This puts higher in-service stresses on the more limited remaining wall webbings between the bubbles, and the remaining thin walls between the series of bubbles can rapidly fatigue and fracture as a result. That effectively allows the voids of the different bubbles to join, forming an irregular, small, wormhole-shaped passageway through the metal, allowing ammonia to escape the tank.

One method to minimize weld porosity is to thoroughly clean, brush, and/or degrease the metal to be welded. However, complete cleaning is sometimes impractical or impossible, and in those cases, welding performed by methods that generate a liquid flux over the metal's surface may resist porosity formation better than gas-metal-arc welding. Some electrodes have metals alloyed

into the welding wire that promote surface cleaning. Filler metals that contain reactive elements such as manganese (Mn), zirconium (Zr), silicon (Si), titanium (Ti), or aluminum (Al) may reduce porosity by having these reactive elements preferentially react with surface residue materials. Ideally, products such as a flux coating react with these metals to remove them by trapping them in a surface slag that generates little or no vapor.

Porosity can also result in arc welds that are protected from the surrounding air by flowing shielding (inert) gas. When gas flow is either inadequate or excessive, contaminated, perturbed by wind or ventilation systems, or prevented from completely covering the weld bead (e.g., welds formed by the so-called drag or back-hand technique in which the welding stick is pulled along ahead of the weld bead rather than pushed in from behind the bead), the liquid metal can absorb oxygen (O_2) and/or nitrogen (N_2) from the air.

Weld porosity can also result from volatile elements within the metal being welded, such as high sulfur (S) content, phosphorus (P) content, hydrogen (H), or the presence of zinc (Zn) on the surface of galvanized steel. However, steels used to fabricate nurse tanks contain minimal concentrations of volatile elements. Therefore, porosity is unlikely to be caused by residual volatile elements in nurse tank steel and thus is an unlikely source of pinhole leaks in nurse tanks.

2.3 FMCSA EXAMINATION OF MANUFACTURER'S WELDING PROCEDURES

At the request of FMCSA, an unannounced inspection visit was made by a senior staff engineer from the National Board of Boiler and Pressure Vessel Inspectors (NBBI) to a facility of the manufacturer. The engineer wrote a report on his observations from the visit and sent that report to NBBI, which provided a copy to FMCSA. The key findings reported were:

- Wet steel surfaces were being welded with no attempt to dry the metal before welding.
- Tank shell courses and heads were being assembled and tack welded without first removing light oxidation scale visible on the metal. The joints were power brushed with wire brush wheels after tack welding, but this brushing could not access the joint at the weld root since the parts were already tack welded.
- Tack welds used on the shell longitudinal seams and the circumferential seams (shell-toshell and head-to-shell) did not have the metal surfaces of their stopping and starting ends properly prepared for incorporation into the final weld.

In addition, the engineer reportedly observed that workers failed to check the rolled shells with the quality assurance template to verify that they had the correct geometry prior to welding. He also observed two complete vessels being assembled without the offset (joggle/flange) area and the edge of the heads or shells being cleaned (to remove dirt or contamination) at the head-attachment station.

Finally, the engineer noted that as a part of the company's material receiving and acceptance procedure, orange paint was supposed to be applied to material to indicate that it was acceptable for use. He witnessed several nurse tank heads without orange paint markings being assembled

on vessel shells; he also noted that orange paint was absent on completed vessels at the hydrostatic testing area.

FMCSA has since concluded its enforcement case with the manufacturer, who fully cooperated and modified its fabrication processes and voluntarily offered extended 5-year warranty protections to all nurse tanks manufactured in 2009 and 2010.

2.4 OUTCOMES OF THE TASKS PERFORMED TO EXAMINE PINHOLE LEAKS IN NURSE TANKS

The following activities were completed during this study's examination of pinhole leaks in nurse tanks:

2.4.1 Acquired five nurse tanks that contained pinhole leaks.

The manufacturer donated three used tanks and a farmers' cooperative group donated two tanks for this project. Data plates for the nurse tanks donated by the cooperative were either nonexistent or illegible. The donated tanks are listed in Table 1.

Serial Number	Size, Gallons	Year of Manufacture	Donation Source
9NF000057	1,000	2005	Manufacturer
DOT S.P. 13554	1,000	Illegible	Cooperative
6NU001139	1,450	2010	Manufacturer
No Plate	1,000	No Plate	Cooperative
9NU001882	1,450	2007	Manufacturer

Table 1. Tanks donated for pinhole leak study.

2.4.2 Performed visual and angle-beam ultrasound testing of donated tanks.

External visual inspection of the pinhole leaks showed a small patch of rust and spalling paint, usually less than 4 cm in diameter around a very small (approximately 1 mm in diameter) aperture that was the pinhole's exit point on the tank's outer surface. The release of pressurized NH₃ has a powerful refrigerating effect as the gas expands and its pressure drops to ambient atmospheric level. Thus, the metal around the pinhole is chilled, which condenses moisture from the atmosphere. This moisture causes the steel to rust. The rusting metal expands, which lifts the paint away from the surface. No other damage was evident on the tanks' exterior surface around the pinholes.

Serial Number	Visible Flaws	Size, Gallons	Year of Manufacture
9NF000057	Leak at end of leg weld.	1,000	2005
DOT S.P. 13554	Pinhole leak next to valve.	1,000	Illegible
6NU001139	Pinhole leak at vapor valve-fitting weld.	1,450	2010
No Plate	Pinhole leak on circumferential head weld.	1,000	No Plate
9NU001882	Two pinhole leaks on roundseam weld.	1,450	2007

Table 2. Visual inspection of flaws.

Angle-beam ultrasound inspection was performed on the region surrounding each pinhole, but this technique was not productive. All the pinholes were in deposited weld metal, and the metal adjacent to a weld is a region that generates strong ultrasound indications whether or not the weld is flawed. Thus, ultrasound examination showed strong reflections along the weld zones, but these were no different from the reflections observed by ultrasound on welds without pinhole defects.

Angle-beam ultrasound inspection was carried out on the rest of these five tanks. These inspections found 19 ultrasonic indications. These were all less than 0.5 inch long and none were within 6 inches of any pinholes (i.e., the pinholes were not part of a crack network).

Serial Number	Flaws Found with Angle-beam Ultrasound	Size, Gallons	Year of Manufacture
9NF000057	Three flaws were found on the bottom of the central circumferential weld under the flange.	1,000	2005
DOT S.P. 13554	Four flaws.	1,000	Illegible
6NU001139	Two flaws.	1,450	2010
No Plate	Five flaws.	1,000	No Plate
9NU001882	Five flaws were found: four on the bottom of one circumferential head weld under the flange, and one on top of central circumferential weld (not under the flange).	1,450	2007

Table 3. Angle-beam ultrasound inspection results.

2.4.3 Cut into the tanks and removed coupons for metallographic study.

This was completed in an effort to determine the shape of the pinhole leaks. Each of the five tanks was cut with an oxyacetylene torch to reveal the interior of the tank and to remove coupons approximately 150 mm on a side for subsequent examination by serial milling and by planned (but not achieved) neutron diffraction.

2.4.4 Performed wet magnetic particle testing of the inner tank walls.

This was done to detect any cracks that might be present and connected to the pinholes. Once the tanks had been cut, it was possible to access the inner surface of the tank and perform wet magnetic particle testing of the inner tank wall. This allowed examination of the inner surface to verify the ultrasound findings of no cracks connected to or radiating outward from the pinhole leak. This testing (see Table 4) verified that there was no correlation or connection between the pinhole leaks and the few cracks that were present on the tanks' inner walls. However, it did find crack networks that were not detectable by ultrasound, because the cracks were less than 0.5 mm deep.

Serial Number	Flaws Found with Fluorescent Wet Magnetic Particle Inspection	Size, Gallons	Year of Manufacture
9NF000057	One 1-inch flaw was detected. This was a through-wall crack at the site of a leaking leg weld.	1,000	2005
DOT S.P. 13554	No flaws detected.	1,000	Illegible
6NU001139	No flaws detected.	1,450	2010
No Plate	One 3/8-inch-long crack in the weld near the center circumferential weld was detected. One 3/4-inch-long crack on the weld on the circumferential end flange was also detected. This crack had not yet grown to the point of reaching the shell wall.	1,000	No Plate

Table 4. Fluorescent wet magnetic particle inspection.

Serial Number	Flaws Found with Fluorescent Wet Magnetic Particle Inspection	Size, Gallons	Year of Manufacture
9NU001882	One head circumferential weld had two 1.5-inch crack networks and one 18-inch crack network. The center circumferential weld had one 2-inch crack network. The other end circumferential weld had one 16-inch crack network and three 1.5-inch crack networks. All of these cracks were in the heat-affected zone (HAZ) next to the circumferential weld.	1,450	2007

2.4.5 Performed serial machining on pinhole coupon.

This was completed in order to measure how the contours of a pinhole cross-section change with depth. One of the pinhole coupons was examined by serial milling, a technique that cuts 25-micrometer thick layers off the metal's surface, allowing microphotographs to be taken of the pinhole's cross-section shape as a series of closely-spaced, cross-section photographs. The external appearance of that pinhole is shown in Figure 8. Figure 9 presents representative photographs from the serial milling operation, spaced at 140 micrometer vertical separations along the pinhole's depth through the steel wall.

The shape of the voids-to-steel interface of the pinhole is very irregular and varies with depth below the metal's original surface. The average diameter of this pinhole was approximately 1 mm, but the diameter varied greatly from one depth to the next as the milling progressed through the full thickness of the tank wall. The varying cross-section shapes and dimensions demonstrated that it was not a simple crack, but an amalgamation of multiple bubbles/voids. This is consistent with the researchers' theory that the through-the-wall pinhole is actually a composite of many porosity bubbles that are later joined together by the process of accelerated metal fatigue failure of the remaining thin steel webbings between the bubbles.



Figure 8. Photograph. Exterior of a pinhole located at the vapor valve (same pinhole is depicted in Figure 9).



Figure 9. Grouped photograph. Metallographic sections of a serially-machined pinhole leak defect.

2.4.6 Transported a pinhole leak coupon section to LANSCE.

This task was completed in an effort to measure residual stresses in the metal surrounding the pinhole leak. A 6-inch by 6-inch coupon with a pinhole leak at its center was shipped to the Los Alamos Scientific Laboratory in Los Alamos, NM for examination of residual stress distributions in the metal surrounding the pinhole using the Spectrometer for Materials Research at Temperature and Stress (SMARTS) detector at LANSCE. This equipment is designed to measure residual stress levels in crystalline materials (in this case the steel surrounding a pinhole leak). It was planned to use this technique to gauge the presence of unusual residual stresses in the steel that might be related to the cause of the pinhole's formation, and that might allow prediction of the probability that a pinhole could grow to a size that might cause catastrophic ammonia release by either stress corrosion cracking or simple fracture.

Unfortunately, the LANSCE beam line was shut down in mid-August of 2012, less than 1 week prior to this project's scheduled examination of the pinhole leak coupon. The shut-down resulted from a spill of radioactive technetium (Tc) by workers performing a project unrelated to the nurse tank safety project. The beam line was not re-opened for research until late January 2013.

This project's beam time was re-scheduled to late February 2013. The LANSCE managers attempted to service as many projects as possible, which they did by reducing the total amount of time originally allocated to those projects for nurse tank research. It should be noted that the nurse tank safety project was fortunate to obtain even this reduced beam time in February, as several projects that had been scheduled to use the beam in the fall of 2012 were denied beam time altogether during the beam's early 2013 restart operation period.

The reduced beam time granted to the nurse tank safety project was used to measure residual stress in a nurse tank manufactured with an ASME protocol post-weld heat treatment (PWHT), leaving no time to measure residual stress levels in the pinhole leak coupon. The next LANSCE beam cycle would not begin until after this project's extended end date.

2.4.7 Assessed findings to determine the cause(s) of the pinhole leaks.

Similar to the damage observed in classic pitting corrosion, pinhole flaws in nurse tanks were found by this project to be narrow channels in the weld metal, but in other regards, they are quite different from classic pitting corrosion damage. Pinholes in nurse tanks often form near the top of the tank around welds for valves and vents. This is inconsistent with the normal downward growth of a pitting corrosion hole on the bottom of a container like a tank. Only rarely does pitting corrosion progress upward.

The serial machining performed (see Figure 9) shows the very irregular shape of the hole through the tank steel. There was no tendency for the channel to widen below the initiation site. In fact, the hole often narrowed to dimensions smaller than one typically sees in pitting corrosion damage sites. The serial machining also showed a more irregular interface between the metal and the hole than one typically sees from pitting corrosion.

Pitting corrosion is most common in the presence of chloride (Cl^{-1}) , bromide (Br^{-1}) , and hypochlorite (ClO^{-1}) ions. None of those ions is expected to be present in significant amounts inside a nurse tank NH₃ environment. Although pitting corrosion can occur in the types of steel used to fabricate nurse tanks, it is more frequently observed in stainless steels. For these reasons, the researchers concluded that the pinhole leaks in the examined nurse tanks did not result from pitting corrosion.

The serial milling and wet magnetic particle examinations showed that the pinhole is not a simple hole or crack, nor is it connected to other cracks in the metal. In addition, as previously noted, the steels, processes, and procedures used in nurse tank manufacture should not cause weld joint cracking. Thus, the researchers also rejected the cold lap and weld crack mechanisms as possible causes of the pinhole leak defects.

2.4.8 Radiographic inspection of a pinhole leak.

This was completed in an effort to confirm the research team's theory regarding the origin of the weld porosity. Tank 9NU001882 was selected for radiographic examination because it had two pinhole leaks in a girth (shell-to-shell circumferential) seam-weld joint (Figure 10).



Figure 10. Photograph. Two pinhole leaks in the roundseam weld of 1,450-gallon tank 9NU001882.

A section of this tank was cut with an oxyacetylene torch and then analyzed at ISU's CNDE. Staff scientists from CNDE took radiographic images in accordance with American Society of Testing and Materials (ASTM) standard 1742-06—"Standard Practice for Radiographic Examination." These images are shown below in Figure 11 and Figure 12.

A pinhole leak that forms in a weld due to the presence of porosity does not generate a characteristic "pinhole" image on a radiograph (x-ray transmission photo). Only the porosity is visible because of the small size of the pinhole channel and because the pinhole channel is nearly perpendicular to the sample surface. However, the porosity that is thought to be the underlying cause of the pinhole leaks is usually discernible on a radiograph, so the presence of porosity on a radiograph is an indication that a pinhole leak could form in that region of a weld.

A line of porosity can be seen nearly following the center of the weld, and this line of porosity "dips" at the site of the second pinhole. This line of porosity along the weld is an indication of a much larger overall weld quality problem. The largest pore has a diameter of 0.085 inch.

There is an additional ingredient at work here. The code issued by ASME in 1998 allowed for the use of thinner steel plates in tank manufacture if a 100-percent radiographic inspection was completed for the longitudinal weld joining the shell of the tank. In 1999, both U.S. fabricators of nurse tanks began using the thinner steel.

At present, the code still requires only spot inspection of the circumferential girth welds, which are 48 feet in each 1,450-gallon nurse tank. If the girth weld of the tank shown in Figure 10 had been radiographically inspected at the point of the pinhole, it should have displayed porosity and thus failed the inspection criteria and not been placed in service.

All nurse tanks are routinely checked by hydrostatic testing as a manufacturing quality assurance step. That means that even though this tank had considerable porosity in the welds, it did not have a pinhole leak at the time of the manufacturer's hydrostatic test. The researchers hypothesize that the pinhole developed rapidly (after the nurse tank was in service) from metal fatigue of the remaining steel membranes between the porosity bubbles. The nurse tank may have been aided in passing the hydrostatic pressure test because the manufacturers used the newer ASME hydrostatic pressure of 325 pounds per square inch (psi) rather than the regulatory specification of 375 psi.



Figure 11. Photograph. Radiographic image of section of center girth weld showing radio-opaque markers (1 and 2) placed at the locations of the pinholes.



Figure 12. Photograph. Close-up of the weld shown in Figure 11.

Note: A line of porosity (ellipses) can be seen in the center of the weld.

2.5 CONCLUSIONS ON CAUSE OF PINHOLE LEAKS

2.5.1 Probable Cause of Pinhole Leaks

In the opinion of the researchers, the probable cause of pinhole leaks in the weld seams of the studied nurse tanks is bubbles or voids (i.e., weld porosity that rapidly interconnected through metal fatigue fractures of the remaining membranes). The multiple deficiencies in material preparation for welding reported by the NBBI inspector would be expected to cause such weld porosity problems, and the irregular geometry of the pinhole revealed by the serial machining technique is consistent with weld porosity. This is supported by the radiographic inspection of a pinhole that showed extensive porosity along the weld line.

The absence of cracks in or near the pinholes is also consistent with this conclusion. Since porosity alone usually will not cause an immediate leak (which should be detected by the manufacturer's hydrostatic testing), the probable cause of perforation is fatigue cracking in the remaining tendrils of steel lying between the voids/bubbles. Voids can carry no load, so the steel remaining between the voids is compelled to carry the load that would normally be distributed over a much larger volume of steel. This overload presumably drastically shortens fatigue life and leads to rapid fractures that produce perforation in service.

2.5.2 Low Likelihood of Catastrophic Failure of the Tank

The researchers further conclude that the likelihood that pinhole leaks will lead to catastrophic failure of the tank is low. The pinholes are so narrow that they are far smaller than the critical flaw size in nurse tank steels described in the Phase 1 final report. This is true even for steels chilled by the refrigerating effect of rapidly escaping ammonia. Thus, tanks that develop pinhole leaks should be taken out of service for repair or replacement, but their pinhole leaks do not pose an imminent threat of catastrophic rupture. However, the structural integrity of other welds on tanks with pinhole leaks may have been compromised by the presence of porosity in the welds.

The extent of such degradation cannot be determined from the data acquired in this study, but an indication of possible SCC problems resulting from substandard welding practices is suggested by the results reported in Section 5.4.

3. STRESS CORROSION CRACK GROWTH MEASUREMENT

The following steps were taken by the research team when performing SCC tests:

- Acquired two additional 500-gallon tanks for use as SCC test bed tanks.
 - Modified the tanks by adding manways.
 - Designed and fabricated SCC test specimens.
- Performed 1-year duration SCC tests in various ammonia solutions and purging conditions.
- After SCC tests concluded, inspected the interior surfaces of the test tanks with NDE wet magnetic particle tests.
- Analyzed the data from SCC testing and developed new plots for SCC crack growth rates as functions of N-Serve content and purging versus no purging during refills.

3.1 THE SCC EXPERIMENTAL PLAN

To measure crack formation and initiation rates in the steel that is now most commonly used to manufacture nurse tanks (SA455), specimens were held in various levels of tensile stress while placed in three different environments:

- A 550-gallon tank equipped with a manway and filled to 80 percent with NH₃ with 0.2 percent water, to determine crack rate behavior.
- A 500-gallon tank equipped with a manway and filled to 80 percent with NH₃, 0.2 percent water plus N-Serve to determine the effect of N-Serve additions.
- A 500-gallon tank equipped with a manway that was vacuum pumped to -15 psi and backfilled with 99.95-percent pure N₂ before being filled to 80 percent with NH₃ containing 0.2 percent water, to see if cracking could be lessened by reducing O₂ content remaining in the vapor space of the tanks.

Each tank had 66–67 samples. One half of the samples were suspended in the vapor region and the other half were suspended in the liquid region of the tank. To facilitate crack formation to get measures of crack propagation rates, single-edge notched tension (SENT) samples were used with a constant load applied via a large spring (as shown in Figure 13).

These 200 test specimens for SCC testing were fabricated in the Ames Laboratory Machine Shop. Sample-holding racks were fabricated by the project staff to position the specimens at the desired locations in the ammonia tanks. Each sample was held in elastic tension throughout the test immersion. The samples were equally distributed in stress intensity from 40 MegaPascal square root meter [MPa(m)^{0.5}] to 95 MPa(m)^{0.5}.



Figure 13. Rendering. Design for constant-load SENT SCC samples.

During June 2011, half of 66 of these SCC specimens were loaded in normal $NH_3 + 0.2$ percent H_2O solution and the other half were loaded in the vapor area above the liquid. During July 2011, half of 67 of the SCC specimens were immersed in $NH_3 + 0.2$ percent H_2O solution containing N-Serve additive, and the other half were immersed in the vapor area. In early August 2011, half of 67 of the SCC specimens were immersed in $NH_3 + 0.2$ percent water and loaded into a tank that was evacuated with a vacuum pump to -15 psi and backfilled with 99.95-percent pure N_2 ; the other half of the specimens were loaded into the vapor area. This tank therefore presumably contained almost no oxygen, since it was both evacuated to a vacuum condition and N_2 was added before ammonia was added.

All specimens in all three batches were periodically removed from the liquid or vapor NH_3 environment to permit measurement of crack formation and growth during the 12-month period of the SCC test. Crack length after 3 months is shown in Figure 14. Figure 15 shows crack lengths after 6 months. No measureable increase in crack length was measured during the 7th to12th months of exposure. Note that the vacuum-pumped and N₂-backfilled tank was exposed for 9 additional months.


Figure 14. Scatterplot. Crack length after 3 months of exposure.



Figure 15. Scatterplot. Crack length after 6 months of exposure.

Three observations can be made from these plots:

- Consistent with what theory predicts, samples in vapor spaces tend to have more cracks and longer cracks than samples in liquid spaces, because there is no water in the vapor area.
- The addition of N-Serve had no impact on the incidence of cracking, but did cause more corrosion.
- The N_2 purge had no impact on the incidence of cracking. This is likely because industry practice is to let the NH₃ purge the oxygen from the tank.

The crack growth in these Phase II tests was lower than expected based on a similar analysis completed in Phase I. A calculation of SCC growth in low-carbon steel immersed in NH_3 was made by Lunde and Nyborg and is shown in Figure 16.⁽²⁾

$$a_y = a_0 + 3.0 \times 10^{-7} K_{IC}^2 \sqrt{y}$$

Figure 16. Equation. Lunde and Nyborg crack length in meters after *y* years.

Where a_y is the final crack length in meters, a_0 is the initial crack length, and y is time in years. For a sample with a constant stress intensity of 95 MPa(m)^{0.5}, the Lunde and Nyborg estimate for growth rate indicates the crack should have grown to 1.35 mm in 3 months, 1.91 mm in 6 months, and 2.7 mm in 1 year. This illustrates that the stress corrosion process is stochastic, because most of the cracks did not grow in most of the samples. Although the highest rate recorded in these tests is smaller than that found in Phase I, the results from both phases are of the same order of magnitude, as shown in Figure 17.

In Phase I, the fastest growth rate estimate was:

$$a_v = a_0 + 9 \times 10^{-7} K^2_{IC} y^{0.5}$$

Figure 17. Equation. Phase I crack length in meters after *y* years.

The highest growth rate from Phase II was:

$$a_v = a_0 + 2.2 \times 10^{-7} K^2_{IC} y^{0.5}$$

Figure 18. Equation. Phase II crack length in meters after *y* years.

The cracks that did propagate stopped growing after 6 months. A key difference between this study and Lunde and Nyborg's was that their samples were exposed to a constantly increasing load while these samples were exposed to a static load.

Figure 21 shows a close-up of one specimen before, during, and after 15 months of exposure. The crack lengthened during the first 3 months of exposure. No further growth was seen after that.



Figure 19. Photograph. Sample #055 before the sample was inserted into the tank.



Figure 20. Photograph. Sample #055 after 3 months of exposure to liquid ammonia.



Figure 21. Photograph. Sample #055 after 15 months of exposure to liquid ammonia.

Growth rate information is valuable in predicting likely failure times for tanks found to contain sub-critical-sized cracks. Many nurse tanks contain cracks, but the cracks are usually too small to pose any immediate safety threat. Such cracks can eventually grow to dangerous dimensions. Information on how fast such growth is likely to occur (from Lunde and Nyborg and from Phase I and Phase II of this study) can guide inspection procedures to assure that cracked tanks are repaired or removed from service when that becomes necessary.

The researchers do not consider the Phase I and Phase II data points to be definitive enough; therefore, one of the recommendations in this study includes details on how to gather more definitive data on crack growth rates.

3.2 WET MAGNETIC PARTICLE INSPECTION OF TEST TANKS

The three small tanks used in this study for the three different environments of NH_3 were inspected before and after the 12 months of testing (15 months for the vacuum-purged tank). Flaws were found in the simple anhydrous plus 0.2 percent water tank before testing began. These flaws had not grown by the end of the study. These flaws were shallow cracks in the shellto-shell circumferential welds that had been partially ground out by the manufacturer. These circumferential shell-to-shell welds differed from all nurse tank circumferential head-to-shell welds inspected in this project, in that the shell-to-shell welds were double-V welds, while all head-to-shell circumferential welds were butt-welds with one plate offset.



Figure 22. Photograph. Magnetic particle inspection of a weld region, before 12 months of exposure to NH₃.



Figure 23. Photograph. Magnetic particle inspection of the same weld region shown in Figure 22, after 12 months of exposure to NH₃.

The interior surface of the tank containing N-Serve had more corrosion product than the other two tanks. Another example of adverse corrosion effects of N-Serve was that the float gage in the N-Serve tank had corroded and broken off, while the other two float gages were still whole (see Figure 24 and Figure 25). Analysis of nurse tanks during Phase I of this project showed that there was little wall thinning due to corrosion from NH_3 containing 0.2 percent water. However, Phase II observations of the test tank containing N-Serve indicate that tanks containing N-Serve should be monitored to ensure that the walls have not become dangerously thin.



Figure 24. Photograph. The rod from the fill gauge had corroded off its fitting in the tank containing N-Serve.



Figure 25. Photograph. The float gauges from the other tanks (not containing N-Serve) were still whole.

3.3 CONCLUSIONS FROM SCC TESTING

- SCC samples in the vapor space above the liquid's surface displayed more cracks and longer cracks than SCC samples immersed in liquid ammonia.
- N-Serve additions did not cause more cracking; however, they did increase the uniform corrosion rate of the tank's interior wall, forming rust (Fe(OH)_x, where x=2 or 3).
- The N₂ purge during tank refilling did not lower the incidence of cracking. In normal service in the U.S., tanks are refilled without using a N₂ purge. Such tanks are probably also very low in O₂ content because NH₃ vapor serves as an oxygen-free purge gas, making a special N₂ purge unnecessary.
- Shallow cracks found in the test tanks' inner walls by fluorescent wet magnetic particle inspection did not grow during the 12–15 months of test exposure to NH₃.

[This page intentionally left blank.]

4. RESIDUAL STRESS MEASUREMENTS IN AN ANNEALED NURSE TANK

The following tasks were performed by the research team in order to measure residual stress in an ASME-protocol stress-relief-annealed (post-weld heat-treated) nurse tank:

- Purchased a tank that was stress-relief annealed after welding following ASMErecommended protocol.
- Performed external ultrasound and visual examination of the stress-relief-annealed tank.
- Cut hoop section from tank for neutron diffraction analysis and performed dye penetrant NDE examination of tank interior.
- Acquired data on residual stress state of annealed tank hoop section at LANSCE.
- Assembled all findings on stress-relief annealing and prepared recommendations for future manufacturing procedures to reduced SCC hazards in nurse tanks.

4.1 RESIDUAL STRESS IN WELDMENTS

When welds are used to join two pieces of metal, the heated regions of the metal expand as the temperature rises, while the cooler regions distant from the weld do not expand. In addition, the steel undergoes phase transformation after refreezing that changes its crystal structure and microstructure. These changes produce volume changes in the cooling metal. All these changes generate very high stresses in the cooled metal that (unless relieved by annealing) remain in the metal from the moment the weld is completed until the tank is retired from service, often many decades later.

The Phase I final report from this research provides details on the regions in the HAZ near a weld's fusion line (actual melting of the metal occurs only in the fusion zone) and why the HAZ has the highest residual stresses. Some regions near a weld retain a tensile residual stress, and other regions retain a residual compressive stress. Tensile stresses are essential for SCC initiation and propagation, so regions with residual tensile stresses are vulnerable to SCC attack.

The literature search performed for Phase I of this project indicated that no nurse tank had ever been evaluated for residual tensile stresses, either by neutron or x-ray diffraction methods. Diffraction methods provide precise information on the spacing between neighboring atoms in a material. As normally applied, x-ray diffraction has a limited penetration capability. Additionally, the head-to-shell joint has a flange that is an extra layer of metal underneath the head. Thus, to see all the way through the metal, it is necessary to use neutron diffraction.

Residual stress can also be measured by semi-destructive methods. One of the most common methods is described in ASTM E837—"Standard Test Method for Determining Residual Stresses by the Hole-Drilling Strain-Gage Method." This method is limited in that it is only applicable to residual stress profiles where the in-plane stress gradients are small, and it only

identifies in-plane residual stresses. Diffraction measurements can measure strain in three dimensions and results in more accurate stress determination.

The head-to-shell circumferential welds in nurse tanks are welds with one side offset. The offset results in a 1–2-inch-long flange on the inner surface of the tank that protrudes into the shell, which thus obscures the inner surface of both the weld and the HAZ for that half of the weld. ASTM E837 requires placement of strain gauges around the region where the stress will be measured and the flange makes this impossible on the inner surface. Strain gauges could be placed on the exterior of the tank, but ASTM measurement would not give accurate measurements of residual stress on the tank interior, which is where the SCC would initiate.

Regions in a metal with residual tensile stresses are distinguished by atom spacings that are approximately 0.1-0.2 percent longer than normal. Regions in a metal with residual compressive stresses are distinguished by atom spacings that are approximately 0.1-0.2 percent shorter than normal. These net differences in spacing are, at most, only a few picometers (10^{-12} meter), so sensitive instrumentation is needed to make such measurements accurately. Neutron diffraction is one of a small number of techniques that provides these capabilities throughout thick sections of metal plate, such as sort used in nurse tank fabrication.

LANSCE provides a neutron beam, supporting instrumentation, and technical staff to assist scientists in making neutron diffraction measurements. Neutron diffraction is analogous to x-raying a material, except instead of a "picture" you get slightly different patterns of waves determined by the atomic spacing.

The pattern changes as stress or compression are applied to the material's structure. Very accurate measurements of differences in patterns emitted are required. By analyzing the wave pattern, the compressive or stress status in the atomic lattice of that sample can be determined. Figure 26, Figure 27, and Figure 28 illustrate the slight differences is wave patterns that must be measured to determine the amount of compression or stress on the sample.



Figure 26. Image. Wave form indicates normal status, neither compressed or stretched.



Figure 27. Image. Wave form indicates compressed status.



Figure 28. Image. Wave form indicates stretched status.

4.2 RESIDUAL STRESS MEASUREMENTS OF UNANNEALED TANKS

In the previous Phase I research, the residual stresses in two circumferential girth welds were measured for unannealed 1,000-gallon nurse tanks (Figure 29 below), one manufactured in 1986 and the other in 1966. Current industry practice, for more than the past decade of nurse tank manufacturing, is not to anneal nurse tanks. Note that the same manufacturers routinely anneal all or almost all other products they fabricate.

The stress distributions shown in Figure 31 are quite similar for the two different unannealed tanks studied using diffraction in Phase I. In each case, the residual tensile stresses (denoted by the positive stress values on the plots) are nearly as large as the tensile yield strength of the steel. Tensile yield strength is the stress value at which the steel begins to permanently deform under

load. If that deforming load is then removed, the steel will not elastically recover its original dimensions.

When tanks are pressurized with ammonia, additional stresses are imposed on the metal such that the yield strength of the metal is exceeded, and the ultimate strength that the metal can hold is approached (Table 5 and Figure 32). Thus, the combination of the high residual stress near the weld and the pressure of the ammonia places that metal at nearly the highest possible tensile stress state, making it especially susceptible to SCC initiation in these regions.

It is significant that the highest tensile stresses are found at the boundary of the weld and the HAZ, not in the weld's fusion zone. This information can guide future inspection methods, indicating that the most productive search for cracks would be performed along the edge of the weld bead looking at both the HAZ and the weld.



Figure 29. Diagram. Hoop weld sections (12 inches wide) were cut from tanks and examined by neutron diffraction at LANSCE.

The size of the hoops (more than 1 meter in diameter) posed challenges for fixtures to hold the large specimen in the correct orientations in the neutron beam (Figure 30), but these problems

were overcome, and extensive data were acquired on residual stress distributions in and near the hoop welds of these tanks (Figure 31).



Figure 30. Photograph. Nurse tank hoop section in the neutron beam line at LANSCE.



Figure 31. Scatterplot. Residual hoop stress distributions measured by neutron diffraction at LANSCE; tensile stresses are positive, while compressive stresses are negative.



Figure 32. Line graph. Superimposition of the maximum residual tensile stress measured at LANSCE (lower horizontal line) with the stress-strain plot for the steel coupons measured from that tank in tensile testing.

Figure 32 above displays several things. First, the top two curves show the stress-strain behavior of steel specimens from a nurse tank wall as the samples were pulled in tensile stress (y-axis) to ever increasing strain deformation lengths (x-axis). Notice that lines for both samples rise to about 375 MegaPascals (MPa) without showing any strain deformation. This is the elastic range where the steel will return to its original shape with removal of the stress. As shown in Figure 32, when stress greater than 375 MPa was placed on the steel, the stress began to cause plastic deformation along the x-axis, and the stress rose to a maximum value near 14 percent on the strain axis. Then the stress value actually began to decrease at higher strain deformations until the specimen broke at the right end of the curve.

The lower horizontal dashed line shows average stress (created by the welding) contained in unannealed, in-service tanks . Note that the stress level is almost at the limit of the elastic range of stress that the steel can manage.

The upper horizontal line shows the sum of the unannealed residual tensile stress in the weld HAZ, plus the stress imposed by pressurizing the tank with ammonia. This is the maximum net

stress on the steel in an in-service nurse tank, neglecting transient stresses associated with towing the tank over rough ground in farm fields. Thus, with each excursion in hot weather to the maximum pressure, the tank is repeatedly exposed to stresses greater than the yield strength, causing some plastic deformation of the steel. This likely contributes to SCC growth.

The total stress in a nurse tank is the sum of the residual stresses from manufacture and the stress from holding ammonia under several atmospheres of pressure. Table 5 presents the magnitudes of these stresses in the three principal directions (hoop, axial, and radial).

Table 5. Maximum hoop, axial, and radial stresses in a nurse tank resulting from internal pressure and from
residual stress in the HAZ near welds.

I. Stress Direction	II. Internal Pressure (in MPa)	III. Residual Stress in HAZ	Percent of Ultimate Tensile Strength of the Steel Due to (II + III)	
Ноор	110 MPa	350 MPa	82%	
Axial	50 MPa	250 MPa	54%	
Radial	2 MPa	23 MPa	4.5%	

4.3 IMPLICATIONS OF THE RESIDUAL STRESS FINDINGS FOR SCC IN NURSE TANKS

The high stresses in the unannealed HAZ measured at LANSCE are not surprising. Other welds have been measured with this neutron diffraction technique, and it is common to see stresses in unannealed HAZ near the yield strength of the metal. This indicates that thermally-induced hoop stresses during welding are high enough to permanently deform the metal by a small amount. Stresses are lower in the longitudinal direction (parallel to the tank's length) and radial direction (parallel to the cylinder's radius).

It is undesirable to have such high stresses in nurse tank steel. These make SCC from exposure to ammonia in the welded nurse tanks more likely, because the tanks are stressed to nearly the highest possible values during their service lives. This stress level then gets worse when the tank is pressurized, which leads to accelerated SCC initiation, and—to a lesser extent—propagation of the cracks.

There is some consolation to be found in the fact that the high residual tensile stresses decrease rapidly to much lower stress values in regions farther from the weld; thus, crack initiation conditions are more severe near welds, but the stresses tending to propagate the cracks diminish with distance from the weld. This is expected to diminish the speed of SCC expansion as the crack grows outward from the weld.

So these findings suggest that crack initiation is probable, but that crack growth beyond the weld joint is less likely. Much like the previous discussion on pinholes, any small through-crack is likely to cause obvious leakage of the tank prior to catastrophic rupture.

Steel (e.g., the SA455 steel now commonly used in nurse tanks) is a high toughness material, and therefore has relatively large critical crack sizes (the critical size is the length of a through-crack that results in instantaneous failure). For a set of assumptions, that size was calculated to be approximately 3.7 inches in the Phase I report.

4.4 RESIDUAL STRESS MEASUREMENTS OF ANNEALED TANKS

American Welding & Tank (AWT) donated an annealed (post-weld heat-treated) 1,000-gallon nurse tank for inspection. This tank had been annealed with the following ASME furnace profile:

- Place tank in furnace.
- Start oven and run the temperature to 800 degrees Fahrenheit (F) in no specific time.
- Increase the temperature from 800 to 1100 degrees F in no less than 1 hour.
- Hold at 1100 degrees F for 20 minutes.
- Decrease the temperature to 800 degrees in no less than 1 hour.
- Shut down the furnace and remove the tank when cool.

Figure 33 below shows a plot of one of the furnace temperature controllers indicating that the furnace temperature held at above 1100 degrees F for 20 minutes. Note that this temperature is from a thermocouple on the tank. This heat treatment will reduce the residual stresses.



Figure 33. Graph. Furnace temperature profile for 1,000-gallon nurse tank (serial #6NF001939).

A girth weld section similar to that shown in Figure 29 and Figure 30 was flame cut with an oxyacetylene torch from this annealed tank, machined to smooth the cut edges and provide a level surface for mounting, and was shipped to LANSCE. There, strain diffusion measurements were taken in three orientations so that the stresses could be calculated in three dimensions.

4.5 FINDINGS FROM NEUTRON DIFFRACTION WORK

The results of these diffusion measurements and calculations of stress are shown below in Figure 34, Figure 35, and Figure 36. Figure 34 shows the effectiveness of the annealing (or PWHT) process. The hoop stress has been reduced from a maximum value of 346 MPa (50 kilopounds per square inch [ksi]) to a maximum value of 118 MPa (17 ksi). This is a 66-percent reduction in residual stress. The axial stress remains the highest stress in the annealed tank, with a high of 166 MPa (24 ksi), but this has been reduced from a high of 251 MPa (36 ksi) in the unannealed tank. These results demonstrate the effectiveness of the ASME-recommended PWHT in relieving the residual stress in a nurse tank.

It is clearly possible to perform more effective annealing by using higher temperatures held for longer periods. But to do so would consume more energy and take longer to perform, so there is a tradeoff between what is sufficient and the cost of doing so. The ASME protocol represents a current, industry-determined, recommended practice. Research could provide more insight into this trade-off.

The following section describes what was found with the angle-beam ultrasound inspection survey of both unannealed and annealed in-service tanks. (Note that the annealing protocol used on the tanks manufactured by Chemi-Trol in the 1990s is unknown.) These survey results indicate the usefulness of annealing in reducing stress levels that drive SCC initiation.



Figure 34. Graph. Hoop stress across circumferential weld in a fully strain-relieved (S.R.) nurse tank manufactured in 2011 (solid lines) overlaid with an unannealed tank manufactured in 1966 (dashed lines).



Figure 35. Graph. Axial stress across circumferential weld in a fully S.R. nurse tank manufactured in 2011.



Figure 36. Graph. Radial stress across circumferential weld in a fully S.R. nurse tank manufactured in 2011.

It is interesting that the maxima for the hoop, axial, and radial stresses in the fully strain-relieved tank all occur around 40 mm from the weld centerline. This is about 30 mm farther left than shown in Figure 34–Figure 36, which are the maxima on tanks not given a PWHT. The neutron diffraction data do not directly explain why the maxima are shifted 30 mm to the left.

It may be attributable to the volume reduction that occurs when a plastically-deformed region of metal is annealed. Figure 32 indicates that steel in the maximum residual stress region must experience some degree of plastic deformation, since the stresses are expected to exceed the yield stress of the steel. When this metal is annealed, it allows the atomic structure to relax some, which will reduce the volume occupied by the cold-worked metal, because it removes the dislocations present in the metal from its plastic deformation.

Dislocations are linear flaws that distort the distances and angles between neighboring atoms, and they have the cumulative effect of making the metal slightly less dense (by a small fraction of 1 percent). When most of the dislocations associated with the cold-worked residual stress maxima are removed by annealing, the metal in that cold-worked region contracts slightly, imposing elastic tensile residual stresses in the metal that had previously lain outside the region with highest residual stress. These new maxima are substantially lower than the previous maxima, so the annealing has reduced the driving force for SCC, but the locations with the largest tensile residual stresses are now relocated about 30 mm further into the shell of the tank.

4.6 CONCLUSIONS FROM RESIDUAL STRESS MEASUREMENT IN AN ANNEALED NURSE TANK

- The ASME protocol PWHT (or annealing) reduced the tensile hoop stress near welds by two-thirds.
 - This is highly significant for SCC risk in nurse tanks because the hoop stress is the highest type of residual stress present in nurse tanks and tensile stress is a primary driving force in stress corrosion cracking. This puts the remaining stress well within the elastic range of the steel.
- Axial residual stress is reduced by one-third by PWHT.
 - Although axial residual stresses tend to be lower than hoop stresses in nurse tanks, they are still significant, so their reduction further enhances the safety benefit from PWHT. Again, this reduction puts the remaining stress well within the elastic range of the steel.
- Performing PWHT on all newly-manufactured nurse tanks would greatly reduce the occurrence of SCC failure in nurse tanks.
 - A representative from each of the two U.S. nurse tank manufacturers was part of the peer review group for this project, and these persons advised the peer review group that it would be feasible to give every tank a PWHT after welding. They also estimated that the cost of PWHT for all nurse tanks produced likely would not exceed \$100 per tank.

- In this study, the ASME protocol for PWHT consisted of at least 1 hour of slow-rising heat from 800 to 1100 degrees F, a 20-minute hold at 1100 degrees F and at least an hour of slowly lowering the heat back to 800 degrees.
 - Hotter and/or longer annealing treatments would be expected to further reduce residual stresses in nurse tanks. The residual stresses would not be completely eliminated even with much hotter and much longer annealing, and the cost of performing a longer, hotter anneal would be greater. The researchers did not look into whether there is literature available on the trade-off between effectiveness and cost.

5. SURVEY OF DEFECTS IN NURSE TANKS

The following tasks were performed by the research team as they worked to complete the survey of cracks in the sample of nurse tanks used in this study:

- Ordered angle-beam ultrasound units with accessories.
- Met with in-service agriculture cooperative tank owners to obtain permission to test their tanks and to arrange a test schedule.
- Performed a search for cracks in the subject nurse tanks at outdoor storage facilities and compiled findings.
- Analyzed survey findings and made recommendations on best methods for inspection.

5.1 INTRODUCTION

The previous research study, "Testing and Recommended Practices to Improve Nurse Tank Safety, Phase I," was initiated in 2008. It involved the examination of 20 nurse tanks ranging in age (from two tanks manufactured in the 1950s to a tank manufactured in 2009). Of those tanks, four were found to have ultrasonic indications consistent with SCCs. A subsequent analysis of sections of those tanks (using fluorescent wet magnetic particle inspection on the inside surface) confirmed the existence and nature of the cracks found by ultrasound.

Based on theory and *ad hoc* observations of the R-stamp operator on the advisory group, it was postulated that most SCC indications in nurse tanks were likely to be found in the vapor area of the tank. Thus the advisory group recommended that FMCSA fund testing of a more representative sample of tanks to statistically establish the validity of theory and limited observational experience. Subsequently, additional funds were approved to conduct larger-scale NDE testing of nurse tanks with angle-beam ultrasound during the summer months when the tanks would be idle and available for inspection. Those funds were awarded in spring 2012, and the testing was conducted from May–August of 2012.

5.2 METHODOLOGY

To ensure that the testing followed an approved industry method that could be replicated in the future, several standards applied to the current nurse tank inspection. These included:

- ASTM E164: Standard Practice for Contact Ultrasonic Testing of Weldments.
- ASTM E273-10: Standard Practice for Ultrasonic Testing of the Weld Zone of Welded Pipe and Tank.
- ASME Boiler and Pressure Vessel Code 2011a Section V Article 4: Ultrasonic Examination Methods for Welds.

• American Welding Society (AWS) American National Standards Institute (ANSI): Guide for the Nondestructive Examination of Welds. (ANSI/AWS B1.10.)

Of these, the most applicable was found to be the ASME Boiler and Pressure Vessel Code 2011a Section V Article 4. The methodology used for the nurse tank survey followed this standard, with one exception. The undergraduate students did not have the number of hours of experience to be certified. This also indicated that if this practice were adopted to address the National Transportation Safety Board (NTSB) recommendation for an NDE examination of all nurse tanks, it would be reasonably practical to create a large number of adequately trained technicians to perform angle-beam testing of nurse tanks. For this study, 2 weeks were taken to train the undergraduate student inspectors.

Unlike hydrostatic testing, ultrasound inspection can be performed on tanks containing pressurized ammonia. All 532 tanks tested for this project contained liquid ammonia.

The ultrasound inspection units used for this study were chosen for their combined ease of use, performance, economy, and durability, based on recommendations from CNDE.

The angle-beam ultrasonic methodology was tested on trial wedges obtained from optima ultrasonic transducers. Two different AWS wedges were tested and used for calibration, one with a "snail" shape designed to "continuously reflect the internal sound beam to, in effect, 'trap' unwanted noise, providing a better signal-to-noise ratio," and one with serrated, "internally machined grooves to reflect and scatter internal reflections." Both of these wedges were sufficient for detecting cracks oriented perpendicularly to the weld.

Final wedge selection was made to optimize signal strength for the geometry of the steel near nurse tank welds. Weld beads are raised above the surrounding HAZ, so a weld bead forms a "stop" that limits how closely the transducer can approach the centerline of the weld. Smaller wedges can be pushed closer to the edge of the weld bead and can thus detect smaller cracks lying right next to the weld bead. For this reason, the smallest wedge size available (0.5 inch) was selected.

During the selection process, trials were run with 45-degree, 60-degree, and 70-degree wedges. The shallower, 70-degree angle wedge (the angle is reported as compared to the surface normal) allowed more complete inspection of the interior half of the weld, but it was rejected because of the reduction in signal strength versus the 45-degree wedge. The geometry of the selected 45-degree wedge still allowed over half of the weld to be inspected from each side.

The 0.5 inch, 45-degree, quick-change wedges were used for all 532 tanks measured (see Figure 37). The quick-change characteristic was important because the areas being measured often contained spatter from the welding, the wedges gradually became worn from being pushed across the roughened surfaces, and the wedges were replaced as they became worn.



Figure 37. Diagram. Schematic of a transducer and a 45-degree wedge.

In order to ensure that the tests conducted were reliable, a series of test blocks were created from 0.25-inch ASTM SA455 steel, which is the steel now used in new nurse tanks and is a similar composition to the steel used in the construction of tanks built before 1990. These blocks had 5/64-inch and 3/64-inch through-holes drilled out to create a reflection point that returned a signal of similar strength to the observed signal strength of a known tank flaw. These calibration blocks were used a minimum of once per tank, when the machine was first turned on, and then at any other times when there was concern for the accuracy of the results obtained. Such a concern might arise, for example, when a tank that was not actively leaking gave indications in a weld of an extremely long crack. In these cases, not only was calibration checked, but the second student examiner also repeated the test to confirm the results of the initial examiner. Many of these occasions involved suspected weld geometry considerations, where the indication was either extremely long or elusive and difficult to find and confirm by both examiners.

The process for calibration was to set the gain of the ultrasonic detector instrument so that signal from the third reflected leg of a 3/64-inch drilled through-hole was between 90–95 percent of the amplitude of the screen. Inspectors would then record and size the length of all indications that were returned with signal strength greater than 50 percent. These gain settings were such that all seven of the test inspectors found all 20 cracks longer than 0.25 inch in the 15 SCC specimens taken from former in-service nurse tanks obtained during Phase I. These tests also showed that this level of amplitude would correspond to a crack depth of at least 1 mm.

A staff member at the CNDE who is an expert in ultrasonic inspection and an Air Force-certified Level 7 inspector (the highest level in the Air Force), trained the undergraduate hourly research assistants who conducted the survey. This staff member tested the students using the test blocks and ensured that they fully understood the requirements for testing, as outlined by ASME Boiler and Pressure Vessel Code 2011a Section V Article 4. Students were then sent out in pairs to conduct the tests. Roughly 2 weeks were spent training, 1 week was spent at the CNDE on the safety hazards of working around NH_3 and in the operation of the testing devices, and 1 week was spent training in the field.

Training involved the use of prepared samples of weld-zone flaws and cracks from the Phase I work in order to gain familiarity with the detection profiles of the various types of features expected in the field. Several days were spent examining samples taken from tanks where the flaws and/or cracks had led to tank failure and leakage. This was done in order to gain experience with using the equipment on the curved, rough surfaces of actual nurse tanks. As a final test, 15 samples were prepared from sections of nurse tanks containing SCCs.

Fluorescent wet magnetic particle inspection was used to inspect the reverse sides of the samples. During this inspection, the location, orientation, and length of the cracks were recorded. Students were then tested to see if they could determine the location, orientation, and length of the cracks using the side-angle ultrasonic instruments on the exterior of the samples. All cracks over 1/4-inch were detected by all students.

When conducting the actual in-field tests, students surveyed all the major welds on the tanks, namely, the three to four circumferential welds (two head-to-shell and one or two shell-to-shell), the longitudinal welds joining the shell together as a tube, the leg welds, and the welds around the lift handles. Students inspected the weld zone on both sides of the weld (using the transducer) to look for indications parallel to and perpendicular to the weld line.

Due to internal geometry of the tank construction, indications parallel to the weld proved the more challenging of the two inspection methods. This is because the design of the weld seam, especially for the head-to-shell, is such that false positives are common. This resulted in a large number of indications located at the center of such welds from the head side. These are most likely related to the actual overlapping steel in the weld and do not constitute a SCC. Note that this limitation is discussed in detail in the results portion of this section.

The length of the crack was determined by moving the transducer from side to side and determining first where the signal was highest and then where the amplitude of the signal dropped below half of this value as the transducer was moved to the right and left of the maximum signal strength location.

Figure 38, Figure 39, and Figure 40 show students performing ultrasonic inspections of typical tanks. One inspector could typically examine one tank in 2-4 hours. One thousand gallon tanks had 50 ft of welds and had to be inspected in two orientations on both sides of the weld for a total of 200 ft of inspection. One thousand four hundred fifty-gallon tanks had up to 66 ft of welds for a total of 264 ft of inspection. Tanks with more flaws took more time to inspect because the length of each flaw had to be determined, and the length and location had to be recorded. Figure 41, Figure 42, Figure 43, Figure 44, and Figure 45 show the form that was used by the students when recording the indications for each tank.



Figure 38. Photograph. Student conducting an examination of a nurse tank.



Figure 39. Photograph. Close-up of inspection of a nurse tank.



Figure 40. Photograph. Close-up of the unit used to conduct nurse tank inspections.

Nurse Tank Inspection Form	Date:	1	/ 2012
ISU Summer 2012			

inspector's name	

Tank #	

SAFETY	Yes	No
Tank is more than 50 feet from NH3 filling station		
No hoses or equipment are attached to tank valves		
Tank is less than 5% full		
5 gallon tank of water is full		
Hose for 5 gallon tank is functional		
Ventiess goggles are worn with no contact lenses		
Long-sleeved ammonia-proof gloves on with a rolled cuff		
6-8 bottle oz water bottle within reach		

Inspection SHALL NOT proceed unless all of the above safety conditions are fulfilled [yes]

Location								
Coop City State								

Equipment and settings					
Flaw Detector	Avenger EZ	Gain setting necessary for reference response between 90 and 95% of full screep bright for a reflection off the flat bettern			
Transducer	5.0 MHz 0.5" dia. TAB 045	hole in a Minature Angle Beam Block.			
Wedge	45°	If gain setting is NOT between 50 and 70 dB, the transducer must be recalibrated.	db		

Manufacturer	City Manufacted In	Year	Serial number / NatL B.D.

Capacity	Length	Diameter	Sheli Thickness	Head Thickness	Head type	Type (S.R.)	SQ ft surface
gallons	in	in	in	in			ft2

Figure 41. Screenshot. First page of nurse tank inspection form.

Nurse Tank Inspection Form ISU Summer 2012

Angle	Bieam in	spection		
Indic	ations	Transducer	Number indications sequentially and report length of ind	ication(s) in inches.
Yes	None	orientation	Indicate position on diagram. Modify diagrams if necessa	ry.
A.Right	Side Fro	nt Head-to-S	hell weld - Transducer beam path alongside or facing into	weld
		1. Head side		
		along weld		—
		2. Head side		
		into weld		
		3. Shell side		
		along weld		
		4. Shell side		
B 1-6		Into were	- II	
D. LEIK	3008 1100		nell weid - Transducer beam path alongside or facing into	WHD.
		1 Head side		
		along weld		
		-		nn
		2. Head side		
		into weld		
		3. Shell side along weld		
		areng mena		
		4. Shell side		
		into weld		
C. Fron	t Leg We	lds - Inspect	the Shell at Legs Welds	
		1. Beam path		ъ и
		along welds		
		Z. Beam into web/s		
		THE R. L.		

/ / 2012

Page 2 of 5

Date:

Figure 42. Screenshot. Second page of nurse tank inspection form.

Nurse Tank In ISU Summer 2	spection Form		Date:	1	/ 2012		Page 3 of 5
Apple Reem	Inconcilion						
Angle Deam Indicat	releatation	Number					
Yes None	orientation	Number					
D Right Rear	Head-to-Shell	weld - Transdu	cer beam r	ath al	oneside or f	acine into wel	4
						8	
	1. Head side						
	along weld						
	2. Head side						
	3. Shell side						
	along weld						
	4. Shell side						
E. Left. Rear.	Head-to-Shell	weld - Transdu	er beam o	ath ak	oneside or fa	cine into web	
	1. Head side						
	along weld						
	Z. Head side						
	3. Shell side						
	along weld						6
	4. Snell side into weld						
F. Rear Leg W	elds - Inspect	the Shell at Leg	s Welds				
							N 1/1
	1. Beam path						
	along welds						

Figure 43. Screenshot. Third page of nurse tank inspection form.

2. Beam into welds

Nurse 1 ISU Sur	Tank Insp mmer 201	ection Form 12	Date:	1	/ 2012	Page 4 of 5				
Angle	Beam in	spection								
Indications		Transducer	Number indications sequentially and report length of indication(s) in inches.							
Yes	None	orientation	Indicate position on diagram. Modify diagram if necessary.							
G.Right Side Center circumferential Weld(s) - Transducer beam path alongside or facing into weld										
		1. front side								



0

Nurse Tank Inspection Form				ite: /	Page 5 of 5						
ISU Sum	nmer 201	12									
Angle Beam Inspection											
Indic	ations	Transducer	Number indication	ns sequent	ially and re	port length of indication(s) in inches.					
Yes	None	orientation	Indicate position on diagram. Modify diagram if necessary.								
I. Left S	ide, Cen	ter circumfer	ential Weld(s) - Tra	insducer b	eam path a	longside or facing into weld					
		1. front side				,					
<u> </u>		along weld	1			2					
		2. Front side									
		into weld	1			2					
		3. Rear side along weld	1			2					
		and a sea	•			-					
		4. Rear side									
		into weld	1			2					
J. Left S	Side Axia	l weld - Tran	ducer beam path a	longside o	r facing inte	o weld					
		1. Upper side									
		along weld									
		2. Upper side									
<u> </u>		into weld									
		3. Lower side									
		along weld									
		4. Lower side									
DIAGR	M-left	Side - Draw	in circumferential a	w leive bru	elds						
Constants.			in circumerentiar.		6103						
		L L									
Front	- (
I											

Figure 45. Screenshot. Fifth page of nurse tank inspection form.

5.3 RESULTS

During summer 2012, a total of 532 tanks were examined. Records from the Iowa Department of Agriculture and Land Stewardship (IDALS) indicate that they inspect 21,522 sets of nurse tank running gear (the wheels and suspension for nurse tanks) in Iowa yearly. These running gear generally hold one or two tanks. Thus, the number of tanks tested by this research project represents approximately 1.3–2.5 percent of the total nurse tank population in Iowa. Note that only those tanks with legible data plates (which display the year of manufacture) were inspected. The distribution of manufacturers is shown in Figure 46, which roughly reflects the market share held by tank manufacturers. At present, there are only two remaining manufacturers of nurse tanks in the United States. Chemi-Trol was acquired by AWT in 1999. AWT was then acquired in 2013 by Quality Steel.



Figure 46. Bar graph. Summary of tank manufacturers and the number of tanks examined.

A summary of the number of tanks inspected as a function of year of manufacture is shown in Figure 47. Figure 48 shows a summary of the number of indications found per tank as a function of year.



Figure 47. Scatterplot. Tanks inspected as a function of year of manufacture.



Figure 48. Scatterplot. Number of indications as a function of year of manufacture.

Note by the symbol indicating tank size (in Figure 47) that there is a clear trend in recent years toward the purchase of larger 1,450-gallon tanks. While numerous side-by-side 1,450-gallon tank pairs were tested in this effort, no tests were made on triple tanks of any size mounted on a single nurse tank running gear chassis. Only single tanks and double tanks were inspected in this study. It is possible that a triple configuration could be physically challenging to inspect with angle-beam ultrasound. Recently, 3,000-gallon tanks came on the market, and reportedly running gear configurations exist for double- and triple-mounted 3,000-gallon tanks.

In the recommendations section of this report, the following issue is raised: while such configurations (a triple-loaded running gear of 1,450-gallon nurse tanks) are not hauling more than 3,500 gallons in a single container, a triple-loaded running gear of 1,450-gallon tanks would be hauling 4,350 gallons. A triple running gear of 3,000-gallon tanks would be hauling 9,000 gallons. Both are far above the 3,500 gallons at which a Hazardous Materials Safety Permit (HMSP) is required.

5.3.1 Effect of Stress Relief

The effectiveness of stress-relief annealing (PWHT) on reducing or eliminating stress corrosion cracking can be seen in Figure 48 as the "notch" visible in the number of indications plotted. From 1991–1998, all of the tested tanks that were manufactured by Chemi-Trol received full-body PWHT. This practice was discontinued in 1999 when Chemi-Trol was purchased by AWT.

For example, 79 percent (82 out of 104) of those stress-relieved tanks had no indications. For those that did have indications, the mean number of indications in those 104 tanks was 0.769 indications, with a standard deviation of 1.3. The standard deviation is approximately 85 percent greater than the mean. Of all the tanks that had critical indication lengths (assuming the worst case scenario that the cracks were through-cracks, which was not the case), only one was found in a fully stress-relieved tank.

For the other 428 tanks without full stress relief, the mean number of indications was 7.55, with a standard deviation of 15.81. The standard deviation is approximately 105 percent greater than the mean. Thus, not only is the mean 982 percent higher than the mean for annealed tanks, the standard deviation is 1216 times higher than the standard deviation for annealed tanks. As is noted elsewhere, this generally means there is a relatively wide dispersal of results among the unannealed tanks. In statistical analysis, such a low mean with a large dispersion indicates a sharply skewed distribution where the majority of tanks have few indications but a small fraction of the total population has a large number of indications.

Phase I established critical stress concentrations for steel at normal temperatures and for very cold temperatures attained when ammonia is able to start leaking through the tank wall. Using a critical stress concentration factor of 85 MPa(m)^{0.5}, there were 55 tanks with indications of critical length; only 2 of those 55 tanks had received a post-weld stress-relief anneal. Using a critical stress concentration of 158 MPa(m)^{0.5}, there were 35 tanks with critical length indications, none of which were post-weld stress-relief-annealed tanks. Note that the tanks with indications of critical length had not ruptured, because that length would be critical only for a through-crack (i.e., the crack had penetrated all the way from the inner wall surface to the outer wall surface). Clearly, none of those cracks were through-cracks, since none of the measured tanks was actively leaking ammonia.


Figure 49. Graph. Number of indications per tank for strain-relieved (S.R.) tanks.

Note: The mean number of indications per tank (6.3) for the entire population of 532 tanks is marked with a horizontal line.

Level	Number	Mean	Standard Deviation	Standard Error Mean	Lower 95%	Upper 95%
Fully Strain- relieved	104	0.769	1.30	0.128	0.516	1.02
Heads Only	428	7.55	15.8	0.764	6.051	9.06

Table 6. Number	of indications in	tanks with o	or without full	strain relief.



Figure 50. Graph. Alternate representation of data from Figure 49.

Figure 51 below also shows the same data displayed in Figure 48, but instead of the year of manufacture, the age of the tank is plotted and a linear regression line fitted to the data. At first glance, this line appears counterintuitive in that the number of indications decreases as the age of the tanks increases. However, the coefficient of determination (\mathbb{R}^2) value for this fit is 0.0394, which is extremely low, implying a high amount of scatter in the data. What is clear from Figure 51 is that age alone is a poor indicator of the number of flaws in tanks. Other factors that should be considered as possibly contributing to the slight negative slope in the number of indications versus age of tanks line include:

- Changes in ASME specifications allowed newer tanks to be built with thinner steel if the manufacturer followed a 100-percent longitudinal radiographic inspection regimen. This thinner steel must carry the same loads as the previously-used thicker sections, so the stress in the thinner metal is higher. That higher tensile stress can affect SCC rates, because tensile stress is one of the main driving forces that cause crack growth. This is explored in Section 5.4.
- Cracks grow over time. If a crack grows to the point that it begins to leak, the tank will either be repaired or taken out of service. Old tanks with leaks (and a high incidence of indications) are more likely to have been removed from service. This means that some older tanks that possibly contain a large number of flaws may have already been selectively removed from the pool by their owners.



Figure 51. Scatterplot. Bivariate fit of indication number (per tank) by age of tank in years.

When only tanks manufactured after 1998 are included in the analysis, then a trend between the age of the tank and the number of indications is apparent. The bivariate fit of the data from the 168 tanks shows a better (but less than ideal) R^2 value of 0.2733. This is shown in Figure 52.



Figure 52. Scatterplot. Bivariate fit of indication number (per tank) by age of tank in years for tanks manufactured from 1999-2011.

Of the 532 tanks inspected, the mean number of indications per tank was 6.25, with a standard deviation of 14.5. In statistical analysis, such a low mean with a large dispersion indicates a sharply skewed distribution where the majority of tanks have few indications but a small fraction of the total population has a large number of indications. This exactly describes the data seen in Figure 48.

This may possibly suggest that the number of indications per tank is also of interest. More research would be needed to make a more specific comment than this. A larger number of indications may point to something, such as manufacturing process control. The number of indications may aid in determining what factors (i.e., age, heat treatment, etc.) have the greatest influence on cracks.

Most tanks inspected around the Ames, Iowa area were either 1,000-gallon or 1,450-gallon tanks. Figure 53 shows that a wide distribution of number of indications was seen for both sized tanks, indicating again that age is not a good indicator of propensity for cracking. In fact, newer 1,450-gallon tanks (following the post-1998 change that allowed thinner steel) even though thicker than 1,000-gallon tanks, have a larger number of indications on average than older 1,450-gallon tanks.

Two types of tank heads were found: elliptical-shaped and hemispherical-shaped. The number of indications per tank for these two populations is plotted in Figure 54, and statistical data are listed in Table 7. Tanks with elliptical-shaped heads had a higher incidence of indications than tanks with hemispherical heads. This is affected by the fact that most 1,450-gallon tanks had elliptical heads, and most 1,000-gallon tanks had hemispherical heads. Since the use of 1,450-gallon tanks is more recent, the majority of the 1,450-gallon units tested had thinner walls than they would have had before 1999, which appears to predispose them to show higher numbers of indications.



Figure 53. Graph. Indications per tank as a function of tank size.



Figure 54. Graph. Indications per tank by head shape.

 Table 7. Mean and standard deviation data on the number of indications for populations of tanks with elliptical and hemispherical heads.

Level	Number	Mean	Standard Deviation	Standard Error Mean	Lower 95%	Upper 95%
Elliptical 2:1	303	8.26	17.0	0.978	6.34	10.2
Hemi	222	3.58	9.5	0.640	2.32	4.84

Note: These data were not recorded for seven tanks.

PWHT of the entire tank had an even higher influence on the number of indications per tank than did head geometry. The number of indications for full-body strain-relieved versus head-only strain-relieved nurse tanks is shown in Figure 49, with statistical data in Table 6. None of the full-body stress-relieved tanks had more than 7 indications, while 1 of the tanks that had only the heads strain relieved had 108 indications. The difference in mean number of indications was nearly 10 times higher for tanks that had not been stress relieved.

In general, metal that has been plastically deformed at a temperature below about 0.4 of the metal's absolute melting temperature (436 degrees Celsius [C] or 817 degrees F for steel) will be work-hardened by that deformation. This makes the metal stronger, harder, less ductile, and more vulnerable to failing by SCC growth. Thus, metal fabricators often include an annealing treatment (holding the metal at an elevated temperature for a period of time) to "relax" the

changes in the metal caused by the plastic deformation. This returns the metal more closely to the condition it was in before that plastic deformation, while preserving the change in shape achieved by the plastic deformation.

In short, annealing "erases" a portion of the changes in the metal's strength and loss of ductility while preserving the part's overall shape change. Nurse tank heads are shaped from flat steel plate by plastic deformation to form the curvature of the head. This plastic deformation causes residual stresses in the metal that are relieved by the heads' subsequent strain-relief heat treatment. However, in tanks that will be given a full-body stress-relief heat treatment after welding, there is no need to separately heat treat the heads after they are formed. Stress relief would be achieved in the heads (as well as all the other regions of the tank) by the full-body stress-relief anneal performed after all welding has been completed on the tank (PWHT).

Representatives of both manufacturers (who were members of the peer review group) observed that the data from this analysis for the benefits of annealing are quite compelling. They were asked by a representative of a large cooperative on the advisory group if his company could specify annealing in orders for replacement tanks. They advised him that yes, his company could do that.

5.4 POSSIBLE DIFFERENCES IN FABRICATION PROCEDURES

The data very strongly indicate that something in the nurse tank fabrication process significantly changed around 1999. Seventy-four percent of the indications (2,104 out of 2,834) were found in the 168 newer, unannealed, thinner steel tanks manufactured by the surviving manufacturers on or after 1999. These tanks made up only 32 percent of the total tanks tested (168 out of 532). This seems disproportionately high. So researchers worked to understand why this was the case.

The known significant occurrence is that thinner tank walls were permitted in tanks manufactured after 1998, if the manufacturer followed a 100-percent longitudinal radiographic quality assurance protocol. Both U.S. companies manufacturing nurse tanks after 1998 reduced their tanks' wall thicknesses in 1999. To the research team's knowledge, nothing else changed in the manufacturers' fabrication procedures. The finding of number of indications by newer tanks suggests that the reduced wall thickness contributed to the sharply increased number of indications in the tanks manufactured after 1998.

Theory states that axial stress and hoop stress in a nurse tank are both directly proportional to the radius divided by the thickness. Thus, the industry's choice to reduce thickness somewhat increased axial and hoop stress. Since these are the two principal drivers of stress corrosion cracking in nurse tanks, theory predicts that one should expect to see more indications in such thinner tanks, and indeed that is the case. As suggested above, annealing could reduce those stresses, thus reducing the number of indications that would develop in such thinner steel tanks.

Of the 2,788 total indications in the HAZ, 31 percent (867 out of 2,788) were found in 19 tanks manufactured by 1 manufacturer in 1999 and 2000, after the practice of full stress-relief annealing was discontinued and thinner steel was used. As discussed earlier in this report, a U.S. nurse tank manufacturer also produced tanks in this time period that developed pinhole leaks.

Researchers concluded that these pinhole leaks were likely the result of poor welding practices that produced porosity in the welds. As a follow-on to this conclusion, researchers worked to determine whether such poor welding practices may have contributed to increased numbers of indications (other than pinhole leaks) in that manufacturer's tanks.

There is a valid engineering basis for correlating increased porosity in welds to higher residual stresses in and near welds. Pores cannot carry any load, so when porosity is present, the amount of steel available to carry loads in the structure is reduced. That has the effect of increasing the effective stress on the metal around the pores.

As stated elsewhere, since 1999 to the present, only two manufacturers were producing nurse tanks in the U.S., and both manufacturers were fabricating tanks with reduced steel wall thickness during that period. Thus, comparison of the numbers of indications in each of the manufacturer's tanks might reveal whether the poor welding practices believed to have caused pinhole leaks may also have contributed to a larger number of indications discovered in that manufacturer's tanks.

However, this comparison is problematic because one company held a dominant position in nurse tank sales around Ames, Iowa during this time period. Of the 168 tanks surveyed that were manufactured during the 1999-2012 time period, only 9 were manufactured by Manufacturer 2. The other 159 tanks were manufactured by Manufacturer 1.

Figure 55, Table 8 and Figure 56 compare the number of indications in tanks manufactured between 1999 and 2012. The average number of indications observed in tanks produced by Manufacturer 2 was 8 per tank, and the average number of indications observed in tanks produced by Manufacturer 1 was 14 per tank. While this difference may appear large enough to draw a conclusion, there is a statistical problem with doing so.

The small number of tanks in the population from Manufacturer 2 makes it challenging to draw statistically valid conclusions from that disparity. The means plus or minus standard deviations for the numbers of indications for each manufacturer overlap substantially, making it reasonably possible that the apparently lower number of indications in Manufacturer 2's tanks could have been mere statistical happenstance and is not a statistically-significant determination for declaring there is a clear difference between the two manufacturers.



Figure 55. Graph. Comparison of number of indications for inspected tanks manufactured after 1998.

Manufacturer	Number of Inspected Tanks Made by this Manufacturer after 1999	Mean Number of Indications	Standard Deviation	
1	158	14	23	
2	9	8	12	

Table 8. Mean and standard deviation for data shown in Figure 55.



Figure 56. Graph. Number of indications for inspected tanks manufactured after 1998.

5.5 EXAMINATION OF LEG WELDS

During the time of the study, a failure in a tank's leg weld occurred near Casey, Iowa. This failure was reported to the research team by U.S. Department of Transportation (USDOT) officials. That report prompted special scrutiny of the data related to leg welds. A plot of the indication data related strictly to leg welds is shown in Figure 58. Of the total number of indications seen thus far, 83 have occurred near leg welds, in a total of 50 tanks. Again, there is a spike in indication data in the years 1998–2000. Thus, while leg welds are susceptible to indications, they are clearly in the minority. Eighty of these were parallel to the weld (96 percent of the indications). This differs from indications found on the rest of the welds, where 85 percent of the indications were perpendicular to the weld.

Research from Phase I of this project indicates the following. ASME offers a number of examples of guidance for how running gear feet may be attached to the tank shell, but none constitutes a specification. Running gear feet failures generally cannot be attributed to SCC, because the NH₃ is on the inside of the tank, which does not reach the leg feet mount welds. However, because these are unannealed welds, there is the possibility of creating a higher stress HAZ through the steel, making the inner surface where the running gear feet are attached more susceptible to SCC. However, failures around these welds seem more likely to be metal fatigue-related from the repeated stresses placed on the tank shell (through repeated transient stresses associated with traversing rough terrain).



Figure 57. Scatterplot. Nurse tanks with indications at leg welds as a percentage of total tanks tested.



Figure 58. Scatterplot. Nurse tanks with indications at leg welds as an absolute number of tanks.

5.6 OVERALL SUMMARY OF THE INDICATIONS

Forty and two-tenths (40.2) percent of the tanks (214) had no indications, and 59.8 percent (318) had at least 1 indication. In these 532 tanks, a total of 3,326 indications were reported. An indication means some irregularity was detected by the ultrasound signal. Indications were seen in the weld itself and in the HAZ next to the weld. Indications in the weld may result from several conditions, namely, lack of fusion, SCCs, and also from the geometry of the weld bead itself.

Indications in the HAZ are more straightforward to explain since they must come either from a flaw in the steel used (such as a pre-existing scratch or crack from steel processing), or a SCC. Indications may be of two types: parallel, where the crack runs parallel to the weld, or perpendicular, where the crack runs perpendicular to the weld.

Of the 3,326 total indications seen:

- 25 percent (832) were parallel to the weld line.
- 72.7 percent (2419) were perpendicular to the weld line.
- 83.8 percent (2,788) were in the HAZ.
- 14.8 percent (493) were located in the weld.
- 1.35 percent (45) were located in various places not characterized as head or shell.
 - Baffle plate attachment point.
 - Surface flaw.
 - Substandard rework with cutting torch causing indication.
 - Surface toe crack.
- 20.8 percent (690) were located in the shell.
- 76.6 percent (2,548) were located in the head.
- 81.4 percent (2709) were located in or around the head to shell circumferential welds.
- 7.37 percent (245) were located in or around the shell to shell circumferential welds.
- 5.98 percent (199) were located in or around the longitudinal welds.
- 2.47 percent (82) were located in or around the leg welds.
- 72 percent (2,127 out of 2,954) of the indications in the circumferential welds were located at or above the 80-percent fill line.

There were 2,788 total indications detected within the HAZ. Of the total number of tanks examined (532), 51.88 percent (276) had no indications in the HAZ, while 48.12 percent (256) had at least 1 indication. The results pertaining to only the HAZ indications are summarized in the next list:

• 14.8 percent (412) were parallel to the weld line.

- 85 percent (2,371) were perpendicular to the weld line.
- 100 percent (2,788) were in the HAZ.
- None were located in the weld's fusion zone.
- 14.5 percent (405) were located in the shell.
- 85.3 percent (2,377) were located in the head.
- 90.7 percent (2,528) were located in or around the head-to-shell circumferential welds.
- 3.6 percent (99) were located in or around the shell-to-shell circumferential welds.
- 5.3 percent (147) were located in or around the longitudinal welds.
- 0.29 percent (8) were located in or around the leg welds.
- 75.3 percent (1,978 out of 2,627) of the indications in the circumferential welds were located at or above the 80-percent fill line.
- 78.8 percent (82 out of 104) of the tanks with full-body strain relief (PWHT) had no indications in the HAZ.

5.7 DISCUSSION

5.7.1 Number and Types of Indications

While a large number of indications exist in tanks, not each indication is related to stress corrosion cracking. As mentioned earlier, indications in the weld itself are difficult to quantify with certainty. Many of these indications are related to the manufacture of the tank and are not flaws, *per se*. Two examples are illustrated below.

5.7.1.1 Example 1

Cutting apart lifting lugs has shown that these regions often have voids between the handle steel and the shell. These voids occur because the tank handles are T-welds that are fillet-welded on each side. Figure 59 shows one of these tank handles. If complete penetration is achieved, the welds on either side of the handle meet and no void results. However, as shown in Figure 60, if penetration is incomplete, a crack can exist between the T of the handle and the base metal of the tank.



Figure 59. Photograph. Example of one type of tank handle.



Figure 60. Diagram. Schematic showing how a third 'leg' response in the acoustic signal can be caused by a gap between the lug and the base metal.

Although this might be considered a flaw since complete penetration has not been achieved, this type of weld does meet ASME Boiler and Pressure Vessel Code because the welds are at least 1/4-inch thick. The relevant diagram detailing how measurements are taken is shown in Figure 61.



Figure 61. Diagram. Figure UW-16.1 (1) adapted from Section VIII, Div. 1 of ASME 2011a Section 1 of the Boiler and Pressure Vessel Code.

5.7.1.2 Example 2

Many indications found at lap welds, such as the head-to-shell, were recorded separately as indications "in the weld" and were classified differently than those from the HAZ. Micrographs taken from regions with these in-the-weld indications show that these readings can occur because of the nature of the joint between the two welded sections (see Figure 62). In these regions, the weld bead does not penetrate to the bottom edge of the overlapping plate. This produces a "corner trap" (circled in Figure 62) where the acoustic signal can be reflected, producing an indication from the unwelded corner and not from a crack. For comparison, Figure 63 shows an example of a weld where complete penetration has been achieved.



Figure 62. Diagram. Schematic with a cross-section of a lap joint showing why a signal is generated at a weld containing a blind corner.



Figure 63. Diagram. A cross-section of a lap joint showing full penetration of the weld bead. Note: This section of weld did not have indications "in the weld."

These in-the-weld indications were seen in the majority of tanks. Figure 62 illustrates why (in tanks where in-the-weld indications were found) they would be detectable from only one side of a girth weld. It was suspected that over 50 percent of the length of the circumferential welds contained "blind corner" regions.

The ASME Boiler and Pressure Vessel Code Section VIII, Div. 1 categorizes this type of joint as a "single-welded butt joint with backing strip" and states that maximum allowable joint efficiency for calculation of the strength of such a weld with no radiographic inspection is 0.65.⁽³⁾

Even with this allowance, the preponderance of tanks with these indications and the pervasiveness of indications on some tanks (over 50 percent of circumferential welds) make this a cause for concern.

Unfortunately, single transducer ultrasound inspection of weld material has inherent ambiguities that make it nearly impossible to clearly identify which one of multiple possible geometries is causing a particular indication. Ultrasonic inspection with phased arrays using many small transducers can provide better images of such indications, but this is an advanced NDE technique.

Because it is not widely in use, it is difficult to estimate precisely the costs of such instruments and inspections. An estimate obtained from an NDE expert is between 5–10 times higher than the cost of performing the simpler side-angle ultrasound inspection. This is because the equipment needed for phased-array inspection is more costly, and its use is more labor-intensive. The simple side-angle ultrasound inspection is expected to prove less costly than a hydrostatic test, but a phased-array test could be more expensive to perform than hydrostatic testing due to its higher equipment cost and greater labor demands.

Because of the safety concerns with the subset of these tanks with false echoes in the welds, some form of additional testing—either hydrostatic or phased-array ultrasonic inspection—is required to determine the safety of the tank. It is relatively time-consuming and expensive to hydrostatically test a nurse tank. This is because the tank has to be emptied of ammonia and

towed to a remote site that reduces or eliminates the possibility of damage or injury if the tank fails during hydrostatic testing.

If phased-array ultrasonic inspection could provide this information, it might be a lower operational cost over the long run than hydrostatic testing, even though the equipment costs more initially. At this point, the additional cost (and thus practicality) of phase-array testing is unknown and would need to be evaluated to determine the performance of phased-array testing before a final recommendation could be made. The simple side-angle ultrasound inspection is expected to prove less costly than a hydrostatic test, but a phased-array test could be more expensive to perform than hydrostatic testing due to its higher equipment cost and greater labor demands.

Indications in the HAZ provide more cause for concern, since the only explanations for these indications are pre-existing cracks or SCCs. These indications are almost certainly SCCs.



Figure 64. Graph. Tanks with critical crack lengths, assuming through-cracking exists.

Thus, while many indications can be found, the process of identifying true flaws is not always straightforward. Things that need to be considered during this process include:

- Location of the indication.
 - Is it found where false echoes are typically found?
- Size.
 - Is it extremely small?

- Direction.
 - How dangerous is an indication in this orientation?

5.7.1.3 Location

Several Pareto plots (Figure 65) have been developed showing the distribution of locations of indications found in the tanks tested, as well as whether the indications are parallel or perpendicular to the welds. These graphs of the data show what types of indications occur most often and where they are located. Nearly 80 percent of the indications were found at the head-to-shell weld, on the head side, and perpendicular to the weld.



Figure 65. Bar graph. Location of HAZ indications in a population of 532 nurse tanks.

5.7.1.4 Size of Indication

Data concerning the length of the indications were collected, but it is difficult to draw definitive conclusions as to the nature of each indication. For example, there were 832 indications that ran parallel to the weld. Of those, 493 were of extremely large length. However, these indications may simply be due to the shape of the weld bead or incomplete weld penetration for head-to-shell joints with protruding flange under one side of the weld, rather than stress corrosion cracking. A strong indicator for this is that some of those indications are many times longer than the minimum critical through-crack size and have not failed. Also, although not required, a number of Iowa cooperatives have adopted a practice of routinely subjecting their tanks (even those with data plates) to a hydrostatic pressure test. As noted previously, the researchers coded these indications separately. Obviously, none of those indications are through-cracks, as a

through-crack results in immediate escape of NH₃, which is readily detected by the odor and discoloration at the point of penetration of the tank wall.

If one assumed a worst case scenario and allowed that the indications recorded in the HAZ did represent through-cracks, then of the 2,788 total indications found in the HAZ, only 93 of them in 55 tanks (10 percent of all tanks) would be of critical length at -70 degrees C, where the stress concentration factor is only 85 MPa(m)^{0.5}. Only 57 indications from 35 tanks (6.5 percent of all tanks) would be of critical length at 20 degrees C where the stress concentration factor is 158 MPa(m)^{0.5}. These results are shown in Figure 64. Note again, this assumes through-cracking is present, which clearly is not the case. Note that in the slightly revised Phase II recommended testing protocol flowchart, there is a recommendation for a maximum indication size that should warrant hydrostatic pressure testing (see Figure 67).

5.7.1.5 Direction

Classification of these indications is complicated by the fact that some cracks were present in branched networks (Figure 66). Where gently curving cracks were detected, the lengths of branched cracks were classified as being aligned with whatever direction comprised the greater portion of the crack complex.



Figure 66. Grouped photograph. Magnetic particle highlighted examples of crack branching in nurse tank SCCs.

5.7.2 Effects of Tank Age, Size, and Thickness

The number of indications observed varied with tank age, tank size, and tank wall thickness. Younger tanks had more indications than older tanks. This is probably due to a combination of factors: all newer tanks since 1999 have thinner steel; more recent tanks are larger, mostly 1,450gallon tanks, while older tanks are mostly 1,000-gallon tanks; and larger tanks have more material and more linear feet of weld, so indications would likely be more numerous in larger tanks. Tanks that were given a PWHT (stress-relief annealing) have far fewer indications than tanks without a PWHT. These data are summarized in Table 9 and Table 10. Of the 104 fully stress-relieved tanks shown in Table 9, 81 were 1,450-gallon tanks, and 23 were 1,000-gallon tanks. This seems to indicate that PWHT more than overcame the greater risk of larger size.

Group	Number	Mean	Standard Deviation	Standard Error Mean	Lower 95%	Upper 95%
1,450-gal	208	11.63	19.65	1.36	8.95	14.32
1,000-gal	220	3.70	9.58	0.65	2.42	4.97
Full S.R.	104	0.77	1.30	0.13	0.51	1.02

Table 9. Number of indications, mean, and standard deviation data on the number of indications for 1,450-
gallon, 1,000-gallon, and stress-relieved tanks surveyed.

ЪТ /	T 11	a n	T 11		. 1
Note:	Full	S.R. 3	= Fully	stress-re	lieved.

The 1,000-gallon tanks have approximately 25.8 ft of welds, and the 1,450-gallon tanks have approximately 33.6 ft of welds, (1.3 times more than the 1,000-gallon tanks). However, the 1,450-gallon tanks have 3.15 times more indications than the 1,000-gallon tanks. Interpreting this difference is confounded by the fact that like the 1,000-gallon tanks, the manufactures are now using thinner steel than they used to on the newer 1,450-gallon tanks. This is discussed above in Section 5.4 above.

To get some insight into this, the research team removed the effect of thinner walls by removing tanks manufactured after 1998 from the analysis. The trends are shown in Table 10.

Table 10. Number of indications, mean, and standard deviation data for tanks manufactured before 1999.

Group	Number	Mean	Standard Deviation	Standard Error Mean	Lower 95%	Upper 95%
1,450-gal	45	8.07	14.64	2.18	3.67	12.47
1,000-gal	216	2.77	4.92	0.34	2.11	3.43
Full S.R.	104	0.77	1.30	0.13	0.51	1.02

As shown in Table 10, the 1,450-gallon tanks manufactured before 1999 had 2.91 times (8.07/2.77) as many indications as the 1,000-gallon tanks. Thus 1,450-gallon tanks are more likely to have indications even when the effect of the reduced wall thicknesses in tanks manufactured after 1998 has been eliminated.

The importance of PWHT is further emphasized by the following. In unannealed tanks, hoop stress in a tank increases as radius/thickness increases. This relationship suggests that one would expect to see increasing numbers of indications associated with the industry trend toward using larger-sized tanks, which means they have a larger radius, depending on whether the thickness of the steel is increased proportionately to maintain a constant radius/thickness ratio. This is

because hoop stress would increase as tank radius increases, if wall thickness were to remain constant. However, 1,450-gallon tanks are generally built with thicker walls than 1,000-gallon tanks (the numbers and ratios have varied over the years), so it is difficult to draw clear conclusions from these observations. There were no 3,000-gallon tanks in the 532 tested, but this could imply they may have even more indications than the 1,450-gallon tanks.

5.8 SUMMARY AND CONCLUSIONS

It is clear from the data collected during the summer of 2012 that full stress-relief annealing plays a major role in reducing the number and severity of stress-corrosion-related cracks. Ultrasonic testing has proven to be an effective way to determine the location and size of potential cracks, although false echoes from weld geometry can introduce a certain amount of uncertainty into the survey. Location and size of the crack should be taken into account when deciding whether a true SCC exists. Every one of the 532 tanks examined in this survey remained in-service after examination. Thus, it would be possible to monitor (relatively inexpensively) the cracks found over a regular time interval (e.g. every 2–5 years) to measure crack growth rates. Such follow-up examinations would also help determine which indications were associated with pre-existing weld defects (which would likely remain unchanged over time) and which are true SCCs (which would be expected to grow slowly over time).

The type of steel used to fabricate each of the 532 studied tanks was not marked on the tanks' data plates, but discussions with representatives from the tank manufacturers indicated that it would be possible to determine what steel was used to fabricate each tank based on information that is given on the data plate (i.e., tank diameter and wall thickness values). If that data were requested and obtained from the manufacturers, it would allow for the determination of correlations between steel composition and the numbers of indications observed.

The following conclusions can be drawn from the analysis completed for this study:

- 1. Ultrasonic testing is an effective method for determining the location and size of potential cracks, but false echoes from weld geometry can introduce uncertainties into those measurements. Location and size of indications need to be considered when determining whether a true SCC crack exists. Ambiguities in interpreting ultrasound indications could be resolved by repeating ultrasound inspection on a periodic basis because SCC cracks would likely grow longer during the time between inspections, while indications caused by false echoes would remain unchanged.
- 2. The survey examined 532 in-service nurse tanks and found 3,326 total indications. Most indications (84 percent) were in the HAZ, but a significant number (14.8 percent) were in the weld-fusion zone. About three-fourths (73 percent) of the indications were perpendicular to the weld line, and one-fourth (25 percent) were parallel to the weld line. Only 21 percent of indications were located in the shell, which is the cylindrical main body of the tank, while 77 percent were located in the heads, the rounded ends of the nurse tank.
- 3. The circumferential welds that join the heads to the shell accounted for 81 percent of documented indications. Of those circumferential weld indications, 72 percent were

located in the vapor space above the 80-percent fill line, a significant and disproportionately large percentage, since only about one-fourth of circumferential weld length lies above the 80-percent fill line.

- 4. Only 7 percent of indications were in shell-to-shell circumferential welds, and 6 percent were in longitudinal (girth) welds in the shell.
- 5. The 168 examined tanks that were manufactured during or after 1999 accounted for 74 percent of the indications found in the HAZ, despite the fact that those tanks comprised only 32 percent of the total tanks. There may be a difference by manufacturer, but this study did not have enough data on the second manufacturer to be statistically certain.
- 6. Full stress-relief annealing (PWHT) greatly reduces the number of indications found in such tanks. Seventy-nine percent of the tanks with full-body strain relief had no indications in the HAZ, and there was only one indication that would require a hydrostatic pressure test under the recommended testing protocol below.

6. SUMMARY OF KEY FINDINGS FROM THE PROJECT AND POSSIBLE FUTURE RESEARCH

6.1 WHAT HAS BEEN LEARNED?

- Pinhole leaks in nurse tanks result from the formation of pores, bubbles, and/or voids created during welding, where the webbing between them can fail due to metal fatigue, thus joining the voids to form wormholes. These flaws can be eliminated by assuring that only clean, dry surfaces are welded.
- Pinhole leaks in a nurse tank are substantially smaller than critical-sized cracks and thus are unlikely to lead to catastrophic tank rupture.
- SCC test samples placed in the vapor space above the ammonia's liquid surface displayed more cracks and longer cracks than SCC samples immersed in liquid ammonia. This is consistent with the theory that ammonia vapor, which does not have water vapor in it, is a more aggressive SCC corrosive medium than liquid ammonia, which contains the required water.
- The process of pumping out the air and performing an N₂ gas purge prior to tank refilling did not lower the incidence of SCCs; thus, N₂ gas purging during nurse tank loading would not lower the risk of SCCs in nurse tanks.
- N-Serve additions to ammonia did not cause more SCCs; however, N-Serve did increase the uniform corrosion rate of the tank's interior wall, forming rust (Fe(OH)_x, where x=2 or 3).
- The ASME PWHT (annealing) applied to a 1,000-gallon tank for this research reduced the residual tensile hoop stress near welds by two-thirds and reduced the residual axial tensile stress by one-third. Hoop and axial tensile stresses are the primary drivers of SCC initiation and growth in nurse tanks, so their reduction (following PWHT) to ranges well within the elastic stress range of steel indicates that PWHT greatly reduces the risk of SCC development in nurse tanks. Thus, performing PWHT on all newly-manufactured nurse tanks would greatly reduce the occurrence of SCC failure in nurse tanks. This is even more critical given the trend toward purchasing larger tanks (which hold larger amounts of NH₃ and thus pose a higher risk of damage or injury in the event of catastrophic failure).
- Ultrasonic testing is an effective method for determining the location and size of potential crack indications.
- A sample of 532 in-service nurse tanks were examined by side-angle ultrasound and found to contain 3,326 total indications. Most indications were cracks, but some may have resulted from weld geometry or non-crack internal flaws. The great majority of indications were located in the HAZ beside the weld, but a significant minority were found in the weld-fusion zone. About three-fourths of the indications were perpendicular to the weld line, and one-fourth lay parallel to the weld line. More than three-quarters of

documented indications were located in the tank heads; only about a fifth of indications were found in the tank shell.

- The circumferential welds that join the heads to the shell accounted for the great majority of indications, and most of those indications were located in the vapor space above the 80-percent fill line.
- The 168 examined tanks that were manufactured during or after 1999 accounted for 74 percent of the indications found in the HAZ, even though those tanks comprised only 32 percent of the total tanks examined.

6.2 WHAT ACTIONS ARE RECOMMENDED BASED UPON THESE FINDINGS?

- Pinhole leaks can be avoided in nurse tanks by assuring that welds are made only on clean, dry metal.
- Nurse tank owners should avoid storing ammonia that contains N-Serve in nurse tanks for long periods of time (i.e., months or years). It is understood that the growing trend is to separately inject N-Serve at the time of application rather than premixing it in the ammonia and dispensing via the nurse tanks.
- Ultrasonic testing is an effective method for determining the location and size (though not the depth) of potential cracks. Ultrasonic testing should be performed on the HAZ and the fusion zones of all nurse tank welds as a routine inspection procedure following the revised inspection algorithm described in Figure 67 of this final report. The minor addition to the recommended protocol from that described in Phase I of this research is highlighted. If inspection of all welds on tanks is deemed too costly, the majority of SCC problems could be identified by inspecting the shell-head circumferential welds above the 80-percent fill line.
- PWHT (annealing) should be performed on all new nurse tanks as a part of the manufacturing process to reduce the occurrence of SCC failure.



Figure 67. Flowchart. Revised recommendation for nurse tank inspection procedures.

Note: Shaded area is the new portion that was not present in the Phase 1 version of this flowchart.

- The industry is moving toward using ever larger nurse tanks. Other countries require nurse tanks to have manways to enable florescent wet magnetic particle inspection from the interior where the SCC originates. Perhaps the U.S. should consider requiring manways on larger tanks, such as the 1,450- and 3,000-gallon versions.
- Tanks for NH₃ that exceed 3,500 gallons require a HMSP. Perhaps single running gear arrangements of multiple nurse tanks that together hold more than 3,500 gallons should require a HMSP.

6.3 WHAT REMAINS UNKNOWN THAT WOULD BE VALUABLE IF KNOWN?

6.3.1 Ultrasound Inspection

- Ultrasound inspection of nurse tank welds can generate indications caused by echoes from weld geometry. An improved ability to distinguish SCCs from weld-geometry false echoes would be useful in predicting tank lifetimes with greater accuracy. Earlier in this report it was mentioned that phased-array ultrasound might be a way to accomplish this, although the initial cost of acquiring phased-array inspection equipment to perform such screening is somewhat high and the overall effectiveness of the phased-array ultrasound testing process has not been evaluated.
- This study performed a survey of 532 used nurse tanks. However, that survey only provided data for a snapshot of each tank's condition in the summer of 2012. It would be valuable to know how the 3,326 indications found in that survey change over time. This would provide real-world verification of the SCC growth rates measured during the SCC testing performed in Phase I and Phase II of this study.

6.3.2 Corrosion Inhibitors/Corrosion Barriers

- Operators of tanks and pipelines used in oilfield applications routinely add corrosion inhibitors to the products being transported and stored to reduce corrosion of steel structures held in contact with petroleum and natural gas. It would be valuable to learn whether any of those oilfield corrosion inhibitors can retard or prevent SCCs in nurse tanks, and whether they would be agriculturally acceptable.
- Several common polymeric materials are known to resist attack by NH₃; however, these polymers are too weak to serve as nurse tank wall materials. It would be useful to test whether these materials could be coated onto the inner surface of nurse tanks to provide an inexpensive barrier to inhibit SCC attacks in nurse tank steel.

6.3.3 Post-weld Heat Treatment

• The ASME PWHT procedure was tested in this study. It consisted of at least an hour going from 800 to 1,100 degrees F, a 20-minute hold above 1,100 degrees F, and at least an hour going from 1,100 to 800 degrees F. If future nurse tank manufacture is to include PWHT, it would be helpful to determine the optimum combination of time and temperature to achieve the most cost-effective residual stress relief.

6.3.4 Effects of Steel Composition on Stress Corrosion Cracking

Two steels—SA455 and SA612—have been used in most nurse tank manufacture. These steels have different compositions and strengths. SA455 contains 0.33 percent carbon, 0.79–1.30 percent Mn, 0.035 percent maximum P and S, and 0.13 percent maximum Si. SA612 contains a maximum of 0.29 percent carbon, 0.92–1.62 percent Mn, 0.035 percent maximum P, 0.025 percent maximum S, 0.13 to 0.55 percent Si, 0.38 percent copper, 0.28 percent Ni, 0.29 percent Cr, 0.09 percent Mo, and 0.09 percent vanadium. SA612 is the stronger of the two steels (ultimate tensile strength = 83–105 ksi; yield strength = 50 ksi). The ultimate strength of SA455 is 75–95 ksi, and its yield strength is 38 ksi. It

would be helpful to know if the SCC behaviors of these two steels differ, and if so, to determine which is more resistant to SCCs.

• Representatives of the tank manufacturers in the peer review group indicated that if they were to receive information from the data plates for the 532 inspected tanks (i.e., tank diameter and wall thickness values), they could provide data on what steel was used in that tank. That would allow for the determination of correlations between steel composition and the numbers of indications observed.

6.3.5 Acoustic Emission

• A more sophisticated ultrasound inspection technique called acoustic emission exists for inspection of pressure vessels. Acoustic emission is too cumbersome and expensive to be a practical inspection method for routine nurse tank testing. However, it could be a very useful research tool to determine whether the hydrostatic testing now routinely performed on nurse tanks that do not have data plates may actually be expanding subcritical-sized cracks in the steel and thereby degrading the structural strength of the tank. It is known that hydrostatic pressure testing imposes a higher pressure than that which normally occurs in tank operations, and thus (as demonstrated for existing unannealed tanks in Section 4.2 for existing unannealed tanks) is imposing stress beyond the elastic range of the tank, causing it to plastically deform, as indicated in Figure 32.

6.3.6 Number of Indications Per Tank

• This research determined that there is a wide variance in the number of indications per tank. The research team postulated that the number of indications per tank may be of interest. It has already been distinguished that PWHT significantly lowers the number of indications that appear in a nurse tank. However, even among the two groups, (annealed and unannealed) there are wide differences in the number of indications. For example, larger numbers of indications may point to something, such as manufacturing process control. The number of indications may aid in determining what factors other than PWHT have the greatest influence on cracks. More research would be needed to make a more specific comment on this.

6.4 HOW COULD THE UNKNOWNS BE DETERMINED BY FUTURE RESEARCH?

6.4.1 Ultrasound Inspection

• Side-angle ultrasound "false echo" indications could be distinguished from SCCs by periodically re-examining the 532 tanks studied in this project. Ambiguities in interpreting ultrasound indications could be resolved by repeating ultrasound inspections on the 532 subject nurse tanks on a periodic basis (every 2–3 years, or, on a more cost-conscious basis, every 5 years) because SCCs would be likely to grow longer during the time between inspections, while indications caused by false echoes would remain unchanged. Such research would also generate a large data set of SCC growth rates for tanks in actual field use rather than in a laboratory environment.

• Side-angle ultrasound "false echo" indications might be distinguished from SCCs by using a phased-array approach for more definitively imaging within welds. Research on how well this might work could be valuable for determining how to interpret indications in welds and to make a recommendation on whether phased-array testing could be used instead of hydrostatic testing for the subset of tanks with false echoes in the weld.

6.4.2 Corrosion Inhibitors/Corrosion Barriers

• It would be prohibitively expensive to use full-sized nurse tanks as test specimens to examine the effectiveness of corrosion inhibitor additions in NH₃ and polymeric barrier coatings on the inner walls of steel tanks. However, small test coupons of pre-stressed SA455 and SA612 steel could be immersed in the three 500-gallon test tanks with manways that were acquired for this study in order to measure the effectiveness of existing oilfield corrosion inhibitors and polymer barrier coatings in nurse tanks.

6.4.3 Post-weld Heat Treatment

• Coupons from joggled weld sections (head-to-shell with flange) designed to replicate the head-shell welds of nurse tanks could be given a range of PWHT times and temperatures to determine the amount of residual stress relief achieved by various heat treatment schedules. These findings could guide decisions for what is the optimum balance of residual stress relief at a reasonable cost.

6.4.4 Effects of Steel Composition on Stress-corrosion Cracking

• Using one or more of the three 500-gallon test tanks with manways that were acquired and modified for this project, small test coupons of SA455 and SA612 could be loaded by dead-weighting in tensile stress at 50–90 percent of their yield stress values while immersed in liquid ammonia or suspended in ammonia vapor. Examination of their crack nucleation and growth responses could guide future steel selection for manufacturers to minimize SCC development.

6.4.5 Effects of Steel Composition and Thickness on Stress Corrosion Cracking

• Provide the manufacturers with information from the data plates and obtain from them information on the type of steel that was used in each of those tanks. Analyze correlations between the steels used and the indications found.

6.4.6 Acoustic Emission

• Acoustic emission testing could be performed on selected test tanks during hydrostatic testing to determine if pre-existing subcritical-sized cracks expand during hydrostatic testing.

APPENDIX A— INSPECTIONS OF TANKS WITH PINHOLE LEAKS

NURSE TANK "DOT SP 13554"

A 1,000-gallon nurse tank with a USDOT Special Permit Authorization (DOT SP 13554) was donated to ISU by MaxYield Cooperative. The data plate (shown in Figure 68) was illegible. This tank had a pinhole leak on a liquid vapor valve, as shown in Figure 69. This leak caused the tank to be taken out of service. A close-up of this pinhole leak site is shown in Figure 70.



Figure 68. Photograph. Illegible data plate.



Figure 69. Photograph. Pinhole leak on liquid vapor valve on nurse tank DOT SP 13554.



Figure 70. Photograph. Close-up of pinhole leak on the liquid vapor valve of nurse tank DOT SP 13554.



Figure 71. Photograph. Same pinhole leak after cleaning the site with Calcium Lime Rust (CLR) remover.



Figure 72. Photograph. Reverse side of same pinhole showing underside of weld.



Figure 73. Photograph. Serial mill picture of pinhole path.

NURSE TANK 6NU001139

The 1,450-gallon tank with serial number 6NU001139 was donated to ISU by AWT. The data plate is shown in Figure 74. Manufactured in 2010, this tank quickly developed a pinhole leak (also on vapor valve), as shown in Figure 75.



Figure 74. Photograph. Data plate for nurse tank 6NU001139, which was certified in 2010 by AWT.



Figure 75. Photograph. Pinhole leak on vapor valve on nurse tank 6NU001139.



Figure 76. Photograph. Close-up of pinhole leak on vapor valve on nurse tank 6NU001139.



Figure 77. Photograph. Close-up of pinhole leak on vapor valve on nurse tank 6NU001139 after being cleaned with CLR.

The weld was not visible from the reverse side due to the geometry of the piece.

NURSE TANK 9NF000057

A nurse tank with the serial number 9NF000057 was donated to ISU by AWT. This tank developed a leak at one of the welds connecting a leg to the tank. The data plate for this tank is shown in Figure 78. This is more likely to be a fatigue failure than a welding problem since the leg weld does not penetrate the shell.



Figure 78. Photograph. The data plate for nurse tank 9NF000057, which was certified by AWT in 2005.

A leak occurred at the base of one of the welds attaching a leg to the shell of the tank. This is shown in Figure 79.



Figure 79. Grouped photograph. Location of leak on leg weld of nurse tank 9NU000057.

Interior wet magnetic particle inspection showed that the crack was 1-inch long in the interior of the tank (Figure 80).



Figure 80. Photograph. The crack from inside the tank under ultraviolet illumination.

Note: The 1-inch-long crack has been highlighted with wet fluorescent magnetic particles. The red dotted outline marks the outside location of the leg.

An oxyacetylene torch was used to remove a 1-ft square segment around the crack. This piece of steel was cut with a wet abrasive saw to reveal a cross section of the crack which was then ground, polished, and etched, as shown in Figure 81. If the crack initiated on the inner surface of the tank, then it started in the HAZ. More likely, the crack appears to have begun next to the weld (a close look at Figure 81 shows that the top of the crack is not next to the HAZ, but next to the weld). This location would have been subjected to tensile loading due to the weight of the tank and load of ammonia filling the tank. This loading would have fluctuated due to transit stresses that could have led to metal fatigue failure. The crack appears to be in two parts, with 90 percent starting from the exterior of the tank and 10 percent on the interior.



Figure 81. Photograph. A cross-section of the tank shell at the end of the leg weld.
Note: The upper left corner is the weld bead that attached the leg to the shell. The top and external side of the tank still has a thin layer of paint. The crack extends from the edge of the weld bead through the thickness of the tank. The tank shell is 6.3 mm thick. There are two sections to this crack.



Figure 82. Photograph. Opened-up weld side of crack in tank shell.

Note: The top side is the exterior of the tank; the bottom side is the interior of the tank. Two distinct sections are seen in this crack.



Figure 83. Photograph. Opened-up crack in tank shell, anti-crack side.

Note: The top side is the exterior of the tank; the bottom side is the interior of the tank river lines can be seen indicating that the crack began at the exterior of the tank and grew inward.



Figure 84. Photograph.Displays how river lines begin as closely-spaced steps which merge into larger and more widely-spaced steps as the fracture progresses.

Source: "Fractography: Observing, measuring, and interpreting fracture surface topography," by Derek Hull, Cambridge University Press, 1999.

NURSE TANK 9NU001882

A nurse tank with serial number 9NU001882 (Figure 85) was also certified by ATW and donated to ISU. This tank had two leaks on the center circumferential weld (Figure 86).



Figure 85. Photograph. Data plate for nurse tank 9NU001882, which was certified by AWT in 2007.



Figure 86. Photograph. Nurse tank 9NU001882 had two pinholes on the center circumferential weld.

Wet magnetic particle inspection found several cracks inside this tank. One circumferential headto-shell weld had one 18-inch crack network (Figure 87) on the head side, and two 1.5-inch networks on the shell side of the tank. The opposing head at the other end of the tank had a 16inch-long crack network on the shell side of the circumferential weld. There was another 2-inch crack network near the center circumferential weld. These flaws were aligned parallel to the weld and 1 inch from the center of the weld. This corresponds to the area of highest residual stress. Although they were long, these flaws must have been quite shallow because they did not show up in the ultrasound inspection.



Figure 87. Image. An 18-inch flaw found on the shell side of the interior of a head-shell circumferential weld.

NURSE TANK #655

Nurse tank #655 (Figure 88) was donated to ISU by MaxYield Cooperative and did not have a data plate (Figure 89). This tank had a pinhole leak along one of the weld seams connecting a head to the shell. This is shown in Figure 90.



Figure 88. Photograph. Nurse tank #655 had a pinhole leak along one of the weld seams.



Figure 89. Photograph. Nurse tank #655 did not have a data plate.



Figure 90. Photograph. This tank had a pinhole leak where the head was welded to the tank shell.

Note: In this picture, the head is on the left side of the seam. The cracks in this picture are in the paint only; the tank steel has not cracked along these lines.

REFERENCES

- 2 Lunde L, and Nyborg, R. "SCC of Carbon Steels in Ammonia Crack Growth Studies and Means to Prevent Cracking." Corrosion/89; [papers of the International Corrosion Forum] held April 17–21, 1989, New Orleans, Louisiana, 98, 1989,pg 98/1-10.
- 3 ASME Boiler and Pressure Vessel Code Section VIII, Div. 1.

¹ MIG Welding (GMAW). "Common Problems and Remedies | Lincoln Electric." Accessed at http://www.lincolnelectric.com/knowledge/articles/content/gmaw.asp.