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Start-up of a New Efficient and Green TNT Manufacturing Process

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Summary

The result of the ATK TNT project has been the construction and start-up of a new flexible explosives manufacturing capability within two years of contract award.

- This facility has extensive safety controls and unequaled environmental compliance in the manufacturing of TNT.

During its very first run the new process made higher purity TNT than has ever been seen before from any previous US production facility.

- Previous operation start-up took a year to make spec TNT

On its third test run, over 10,000lb of Mil Spec TNT was manufactured and packaged. This TNT was 100.00% pure by gas chromatographic analysis.

	New Process	Old Process
TNT Purity	100.0%(nominal)	99.8%(Max)
Air Emissions	NOx 0.2lb/hr CO <2.5lb/hr	NOx 166lb/hr CO 110 lb/hr
Red Water	None	8500 gal/day
Non-spec. TNT	0	Up to 10%

Background

Objective:

Design and build a TNT manufacturing plant per proposal to US DoD

- **Safe, Green Process**
- **Flexible Design for manufacture of other energetics**

Requirements

The US DoD required a flexible energetics facility capable of making up to 15Mlb/yr of Mil Spec TNT.

The user requires TNT to fill ordnance in existing equipment and obtain high quality fills. This requires consistent purity (set point) and flake size (thickness and max dimension) product.

Environmental requirements constrained the emissions of waste and by-products (red water, CO, VOC, NOx and SOx, waste water (acidity and organic content), and steam usage.

Approach

Obtain VOC - Use IPT and 6 sigma tools throughout design, construction, start-up

Trade Studies

- Search for best practices
- Obtain data on potential improvements
- Use data based decision analysis tools to select optimum solutions

Use formal PDR, CDR, PRR processes

Detailed Hazards Analysis

Select and train technical operators to drive quality in production

TNT Chemistry

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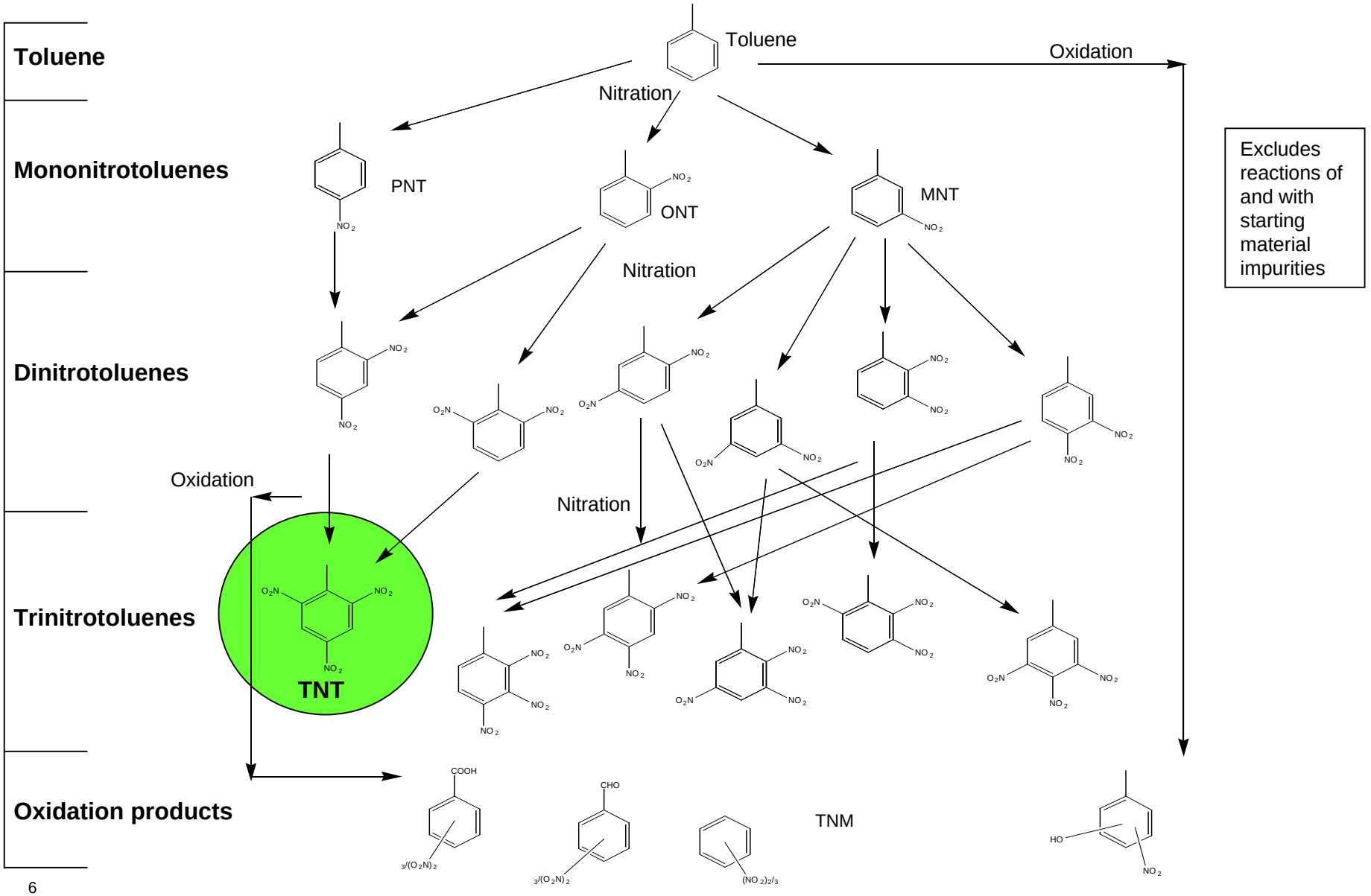

TNT Manufacturing Background

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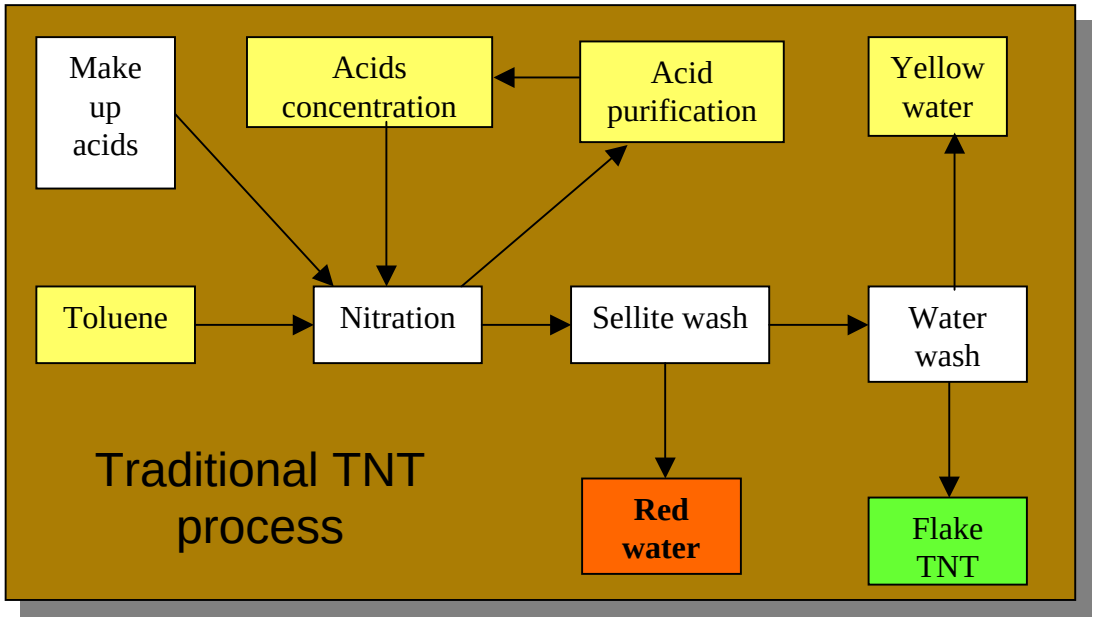
Traditional TNT manufacturing is extremely “dirty”

Waste from traditional TNT production has severe environmental consequences

- K047 prohibited from land disposal 40 CHR Ch. 1 §286.33
- Yellow water
- Air emissions, TNM, CO, NOx, VOC

Alternatives have been used historically that could be employed to avoid some issues

- Purification by crystallization
- Yellow water minimization
- Alternative starting material
- Batch nitration



TNT Trades Relationships

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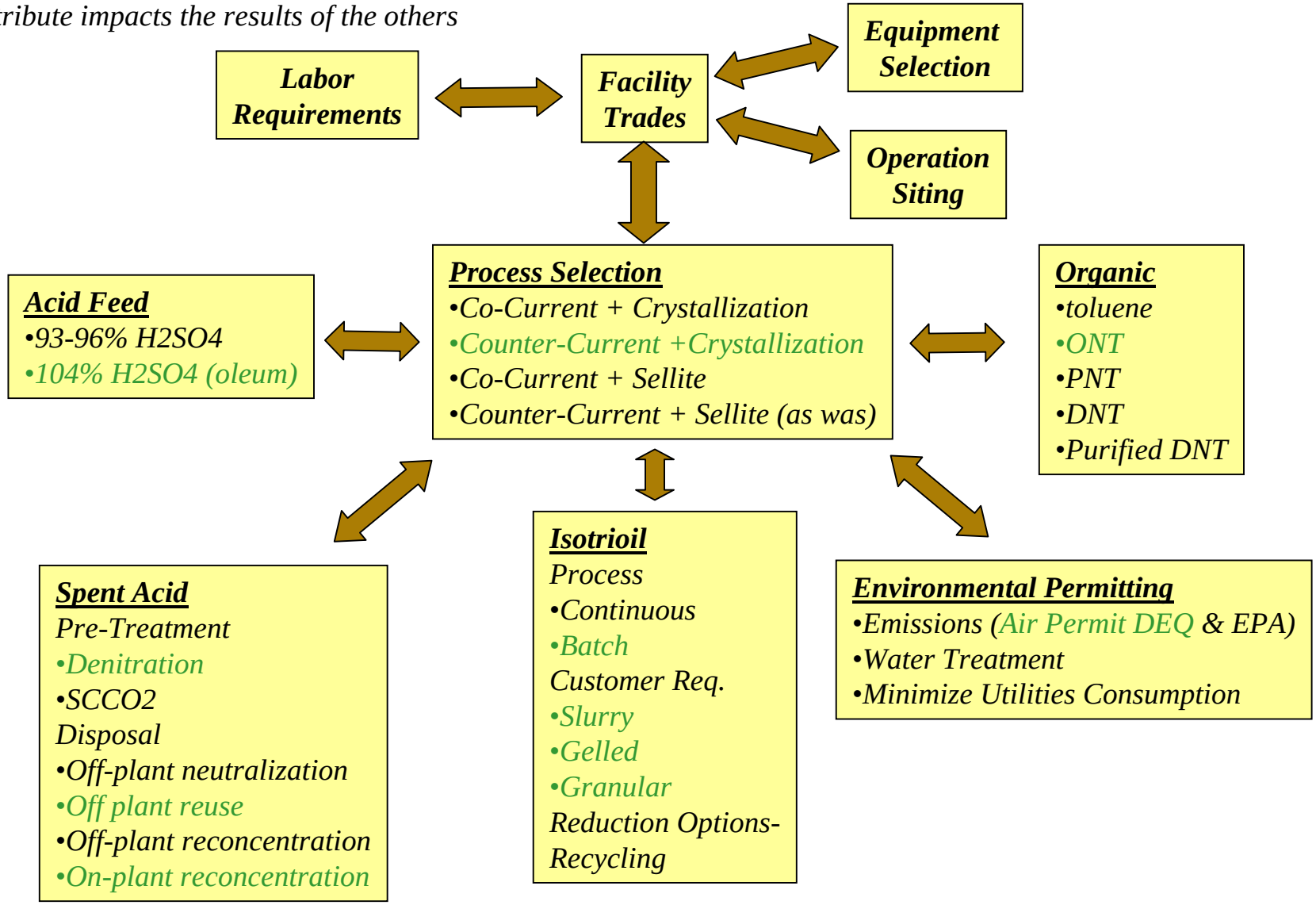
Each attribute impacts the results of the others



Trades Methodology

Tabulate options with detailed analysis, including

- Safety
- Environmental
- Capital and recurring costs
- Impact on other processes
- Technical, schedule, and cost risks
- Human factors

- Use Pugh tables and weighted Pugh tables as appropriate
- Communicate with IPT and customer

Option	Denitration	CO2 extraction	Spent to ..	Notes
1	Yes	Yes	RFAAP NAC/SAC then for sale	Limited NAC/SAC capacity
2	Yes	No	Dupont	
3	Lmited	No	AP	With DNT use
4	Yes	No	GEA	
5	Yes	No	GEM	
6	Yes	No	RFAAP NAC/SAC then for sale	Needs low organics level from Deni

Overall Process at CDR

Starting materials received by rail (truck is also possible) with significant storage possible

Nitration and purification

- Counter current, ONT, anhydrous mixed acid, nitric acid crystallization
  - No red or yellow water

Product finishing, as previous drum flaking with new automated boxing

Isotrioil (TNT impurities) to separate flexible batch treatment and pack out facility

Vapor and spent acid to new denitration and air treatment facilities

Scrubber and similar water to dedicated waste water treatment then plant biological waste water treatment

Process monitoring, quality control and process improvement from control room at area laboratory with chemists running new laboratory, PLC and main operations

Process Controls at CDR

Siemens PCS7 System implemented for TNT manufacturing control

Fiber Optic network connects all manufacturing and support nodes

Nitration and Purification, Spent Acid Denitration & Fume Abatement monitoring and control from two locations

Modernized, automated TNT finishing and pack out controlled locally, with data linked to PCS7 system

Complete data acquisition included (historical trending, SPC charting and integration with LIMS for lab data)

Expansion capability for future energetics manufacture

ACCOMPLISHMENTS

Accomplishments

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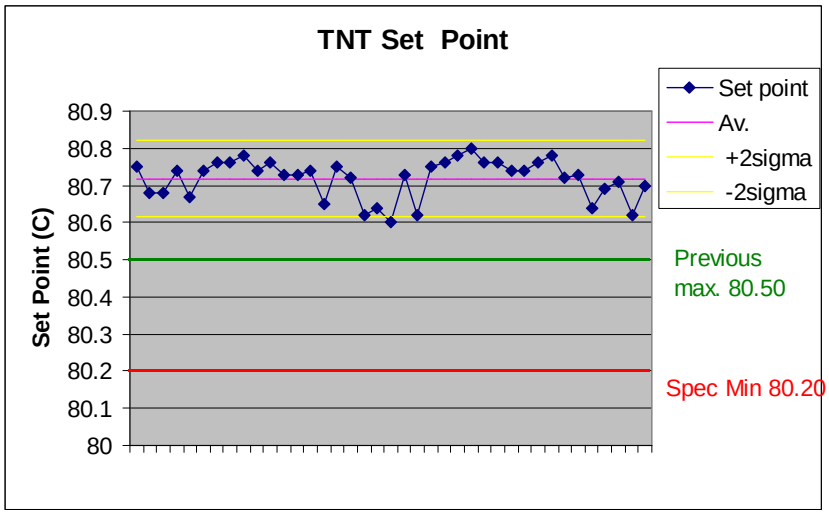
CDR complete in 9 months from contract award

Facility constructed and operational 2 years from contract award

- –>1000 process I/O, >150 interlocks
- –Chemical sampling on every vessel and line
- –Unique solutions to air emissions



Provided lab data for Hazards Analysis

Built and equipped a laboratory

- –Recruited and trained staff; developed, documented, and validated methods

Currently in Preproduction Initial Production

Integration of operations and technical staff to drive operation efficiency and quality

Still discovering improvements

Operations per Building

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ONT in

Isotrioil 9505

A mixture of original buildings with new equipment and entirely new buildings/operations

N&P 9501

Finishing 9504

Pack out

Acids in

Denitration

CO/TNM destruct

Laboratory/control room 9562

Maintenance

Area Office

Nitration and Purification

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Sample points at every vessel (unless under vacuum) and line

Each sample point has nominal values for composition with max and min

Each sample point has a sampling procedure and test procedure for the sample

Troubleshooting guide for out of nominal process conditions

PLC Control and Alarm System

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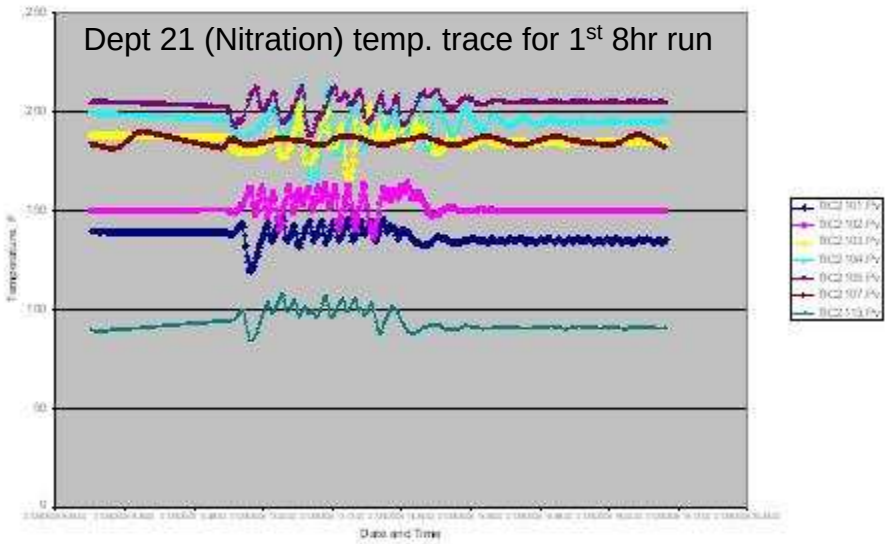

The PLC controls N&P by monitoring raw material flow, speeds, pressures, and temperatures

Upset conditions instigate an alarm on PLC in corresponding department (process)

Emergency conditions sound an alarm and an alert on PLC screen

Multiple levels of alarms:

- Vent system interlocks:
  - high—stop raw material feed
  - high high—increase cooling water supply to jacket of vessel
  - high high high—immediate dump/drown

Emission Comparisons

The New facility has >50x reduction in emissions to air

The crystallization completely eliminates red water emissions with a much more robust purification process

Yellow water is eliminated by reuse in the nitration process before integration in the spent acid (reducing organics in the spent acid at the same time)

Emissions (TPY)	Old Facility	New Facility	Delta (% reduction)
NOx	633.6	0.75	632.8 (99.9%)
CO	416.7	10.95	405.8 (97%)
TNM	40.6	0.83	39.7(98%)

TNT LAT and Process Testing

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Type I TNT Specifications (MIL-DTL-248D)

Lot Acceptance Testing according to MIL STD 650

Form – Flake

- Visual, subjective

Color - No darker than FED-STD-595 Color Standard No. 30257

- Visual, subjective

Solidification Point, ºC - 80.2 min

- Manual measurement

Moisture, % - 0.10 max

- Automated Karl Fischer titration

Acidity, % as H_2SO_4 - 0.02 max

- Manual titration with caustic to a bromothymol blue endpoint after organic dissolution and aqueous extraction

Alkalinity - None

Insoluble Matter, % - 0.05 max

- Gravimetric with organic dissolution and filtering

Sodium, % - 0.001 max

- Atomic absorption spectroscopy (FAA)

Flake Thickness, inch - 0.04 max with an average of 0.025 max

- Manual measurement with micrometer

Flake Size - 100% passing through 3/8" mesh sieve

Six samples tested per day + One 5-Sample Composite

Sample Schedule determined by Mil Spec

In-Process Testing in the Laboratory
30 Routine Samples per Day

	Sample Point	Test
1/nitrator/shift	2101A-5A	Acid, H2O, Organics
AcidWashOF to Cryst	2107A	Organics
AcidWash	2107B	Acid, H2O, Organics
Extractor	2113A	Acid, Organics
ONT/DNT Tk	2117	HPLC
AcidMake-Up Tk	2130	Acid
WaterWashedTNT	2316	HPLC
Acid Tanks	T2318-20	Acid
WNA	2322, 2324	Acid, Organics
YellowWater	2401B-03B	Acid, Organics
Isotrioil	Product	HPLC
Isotrioil	WasteWater	HPLC
Denitration	H2SO4	Acid, Organics
Denitration	HNO3	Acid, Organics
Denitration	Condenser	Acid, Organics
Denitration	Reflux	Acid, Organics
Fume Abatement		Acid, Organics

In-Process Testing in the Production Area
42 Routine Samples per Day

	Sample Point	Test
1/nitrator/shift	2101A-5A	Visual(Acid/Org)
1/nitrator/shift	2101B-5B	Visual(Acid/Org)
1/nitrator/shift	R2101-5	Visual(Acid/Org Ratio)
AcidWashOF	2107A	Visual(Acid/Org)
AcidWash	2107B	Visual(Acid/Org)
AcidWash	T2107	Visual(Acid/Org Ratio)
Extractor	R2113	Visual(Acid/Org Ratio)
Extractor	2113A	Visual(Acid/Org)
Extractor	2113B	Visual(Acid/Org)
PreCrystalizer	T2201.02	Visual for Granules
TNT Washers(YW)	2401A-03A	Visual for YW/Organic
TNT Washers(YW)	2401B-03B	Visual for YW/Organic
TNT Washers(YW)	T2401-03	Visual YW/OrganicRatio)
Emulsion Tank	T2410	Visual (H20/OrgRatio)

Process control/monitoring samples points, sample schedule, testing time, and operating limits identified and methods developed

Chemical Data from First 8hr Run

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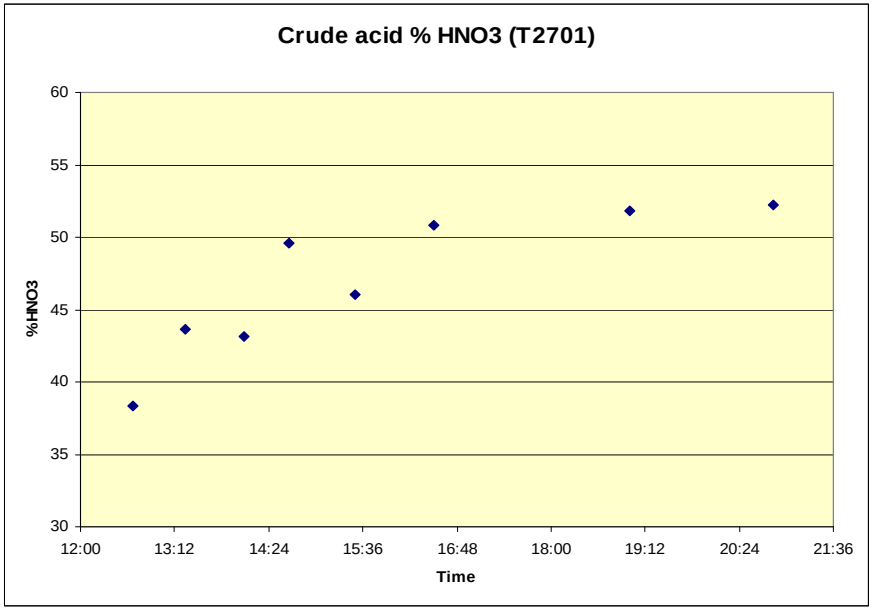
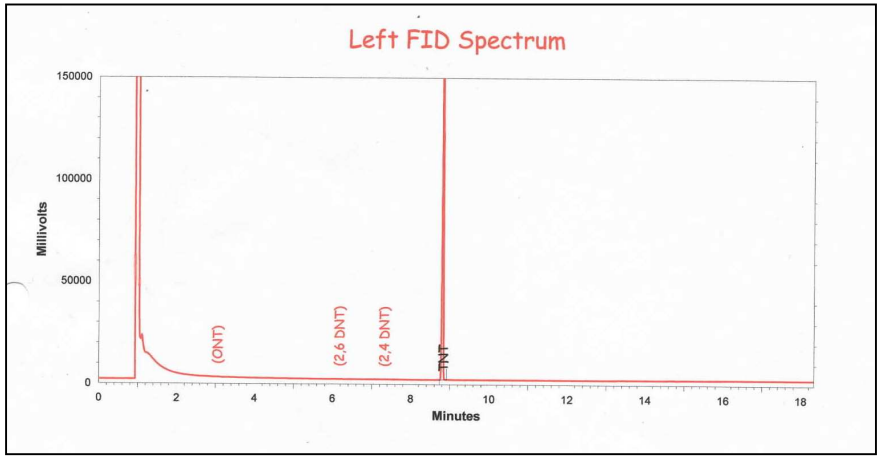
Routine, detailed near real time TNT chemical process data has never been part of production before
TNT of this purity has never been available in the US
The product being pure does not mean the process is perfect

Vessel/line	Time	NB	TNT	24DNT	26DNT	ONT	TNM	Others	SA	NA	NS
R2101	9:40	1.16							67.57	6.31	3.8
R2101	12:20		7.33	23.23	9.86	59.58	0				
R2101	23:59		4.128	23.616	7.326	64.93			65.59	7.456	2.53
R2102	20:35	6.78							81.55	5.42	2.66
R2102	12:20	5.87							78.45	9.38	4.01
R2102	12:20		29.02	42.92	28.07	0	0				
R2102	23:59		16.096	60.3	23.604	0			81.876	3.861	3.401
R2103	18:35	8.54							85.75	8.86	2.65
R2103	12:20	11.7							84.31	8.39	2.5
R2103	23:59		62.761	29.57	7.668	0			86.198	1.019	2.833
R2103	15:10	4.38							72.51	9.09	1.52
R2104	20:35	5.96							89.98	7.26	1.63
R2104	23:59		93.395	5.843	0.762	0			90.904	4.348	2.354
R2104	15:10	6.65							87.05	9.84	1.75
R2105	18:45	12.3							92.27	7.42	0.76
R2105	17:30		98.8	1.2							
R2105	23:59		99.073	0.927	0	0			97.298	7.463	1.225
R2105	15:10	8.93							87.64	9.04	0.9
SV 2115 (extractor SA)?	12:20	1.72	8.1	62.86	26.4	2.63	0				
SV 2115 (extractor SA)?	12:45								62.85	11.55	2.35
SV2113A (extractor acid)	12:20	1.67 (0.93 by	8.04	63.11	26.6	2.25	0		63.78	10.29	2.53
SV2113A (extractor acid)	14:45	0.105 (ext)							64.55	8.64	3.18
SV2113A (extractor acid)	14:45	0.95	6.24	44.37	16.12	33.28					
SV2113A (extractor acid)	18:35	0.79							72.8	5.68	5.09
T1945	15:15	0.27	5.3	29	63.6	2.1	0.02			82.7	7.81
T1945	11:00									71.3	3.57
T2001	?	0.3							68.51	8.12	3.24
T2001 (old method)	25-Jan	0.36							68.69	7.79	4.34
T2001 (withLC)	25-Jan	1.53							69.51	8.11	4.39
T2107 (acid washer) conte	17:30		99.1	0.9			0				
T2107 (acid washer) conte	17:00		98.43	1.31	0.17						
T2318 (xtallization acid)	17:50									48.4	
T2320 (xtallization acid)	20:55									56.6	
T2401 (washer 1)	13:50		100								
T2402 (washer 2)	20:40									0.06	
T2701	20:50	2.47	75.87	21.39	2.74		trace			52.26	
T2701	13:20	1.3	72.99	27.31	3.92	2.33	0			43.62	
T2701	14:05	2.6	66.1	29.8	4.1		trace			43.1	
T2701	14:40	1.88	74.51	19.65	3.14	2.69	0			49.62	
T2701	19:00									51.79	
T2701	15:30	1.95	77.54	17.56	2.67	2.22	0			46.02	
T2701	16:30	2.07	74.146	19.83	3.18	2.84	0			50.84	
T2701	12:40		[illegible]			2.01	trace			38.34	
TNT flake (gc)	9:01										
TNT flake (gc)	18:00										
TNT flake (gc)	17:00										
Washed Isotrioil	26-Jan						est pks @ 0.5				





TNT Production Optimization

Chemical elements to Optimize the TNT Production Process.

- Product purity – throughout process
- Yield based on ONT
- Efficiency based on all chemical reagents
- Throughput
  - Yield per unit of time and labor
- Minimize waste
  - Materials
  - By products
- Robustness
  - Doing all of the above all of the time
  - **All Models and Plans must be Based on Detailed and Accurate Process Data**

The more you understand the chemistry of the production process the safer and the more efficient you can make it become