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Removal of N-Methyl Pyrrolidone From Metal Parts Using A Centrifugal Dryer

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INTRODUCTION

N-methyl pyrrolidone (NMP) is an organic solvent that can be used for the solvating and removing of soils from metal sufaces. With some changes in manufacturing and cleaning processes, NMP can be utilized as a non ozone depleting replacement for 1, 1, 1 Tri Chloroethane in degreasing applications.

The process that is required. for efficient metal cleaning, when NMP is the cleaning solvent, consists of a multi-step immersion process, followed, in some instances by a drying step. Initially, the metal parts are immersed into a heated bath of NMP. This is where the actual soil removal/degreasing occurs. After removal from the cleaning bath, the metal parts are placed into either a single or multiple set of rinse baths. The "oil tainted" NMP is removed from the metal parts during the rinse step (s).

Once the metal parts are removed from the final rinse, the next step that the parts will go through in their own process of being manufactured, will determine whether or not the final rinse liquid will have to be quickly and efficiently dried off of the parts.

This set of experiments was carried out to gather some data on the ability to dry NMP off of bulk volumes of metal parts, so that manufacturers wanting to use NMP, as a 1, 1, 1 Trichloroethane replacement, would have a starting point for drying process development for those instances that NMP is used as the final rinse liquid.

DISCUSSION

NMP is highly miscible with most oxygenated solvents. This allows for the possibility of using many different liquids as final rinse solvents in a metal parts cleaning process. Alcohols, Ketones, water or NMP itself, would be possible final rinse candidates.

Alcohols or Ketones are used in those applications where complete drying is desired in a quick time frame. This usually requires that the alcohol or Ketone bath is heated, so that once the part is removed from the final rinse bath, the liquid evaporates very fast at room temperature. The draw back, of using the hot alcohol or Ketone rinse, is that the flammability properties of hot alcohols are Ketones demand specific safety precautions be built into the rinse station. These safety precautions drive up the cost of the rinse station.

Water is an excellent final rinse liquid for removing trace amounts of NMP from parts, in a cleaning process. NMP and water are totally miscible. For those applications where there is no concern for oxidation, on the surface of the metal, interfering with further processing steps, water is the best choice for a final rinse. Most dryer manufacturers and oven manufacturers are familiar with the physical requirements for efficiently removing water from metal surfaces.

When water or alcohols/Ketones are not options, as the liquids in the final rinse bath, NMP can be used. NMP can evaporate completely from the surface of the metal without leaving a film or residue.

NMP has a slow evaporation rate. So, in order to accomplish the removal of NMP from metal surfaces, in a timely manner, the use of forced hot air is required.

<u>DESCRIPTION OF EXPERIMENTS:</u>

PART 1: DRYING APPARATUS (See Figure 1)
A centrifugal dryer was chosen to dry the metal parts. The specific dryer was a

Turbo 1212
Nobles Manufacturing, Inc.
645 East 7th St.
St. Paul, MN 55106

The dryer was modified in the following manner, so that exact temperature control and air flow could be monitored throughout the test.

a). Turbo 1212, that was used for the test, had (2) heater coils located inside the lid that closed down onto the top of the cylinder where the parts being dried are kept. This heater system has a 4kw capacity.

A thermocouple was attached to the dryer so that it could measure the air temperature just below the heater coils (Thermocouple was placed 0.75 inches below the coiling fins that were part of the heater coil construction approx. 1.50 inches below the actual heater coil surface itself).

b). The thermocouple was tied into a variable heat control unit that allowed for the close control of the output of the heater coils.

The variable heat control had a digital read out. The unit could also be pre-set at desired temperatures.

c). The air exit port at the bottom of the Turbo 1212 unit has a diameter of 3.45 inches. A galvanized steel expansion fixture was clamped onto this exit port. The fixture expanded the outlet pipe diameter to 6".

d). Attached to the galvanized steel expansion fixture was a 2.5 ft. section of 6" diameter flexible duct.

DUCT*: UNINSULATED FOIL DUCT
Meets NFPA 90A
ARP Product TYPE II
ATCO Rubber Products
Fort Worth , Texas

* When duct is referred to in other sections on the modification of the dryer, it is of the same construction.

0.5 feet from the end of the galvanized steel expansion fixture, a port hole was cut into the duct so that temperature and air flow could be monitored at the same position for the duration of the test. (approx. 8 inches from Exit of Drying Chamber)

e). Attached to the end of the 2.5 ft. duct (the end away from the dryer) was an exhaust fan. The fan was arranged so that the air entering the unit was pulled through the centrifugal dryer and exhausted out the other side of the fan.

<u>FAN:</u> Model 4C447
Dayton Electric Mfg. Co.
Chicago, IL
1610 RPM/115 volts
$HZ^{50}/60$ HP $^{1}/20$
Type U6381 #7063-3277
Class B Ins.

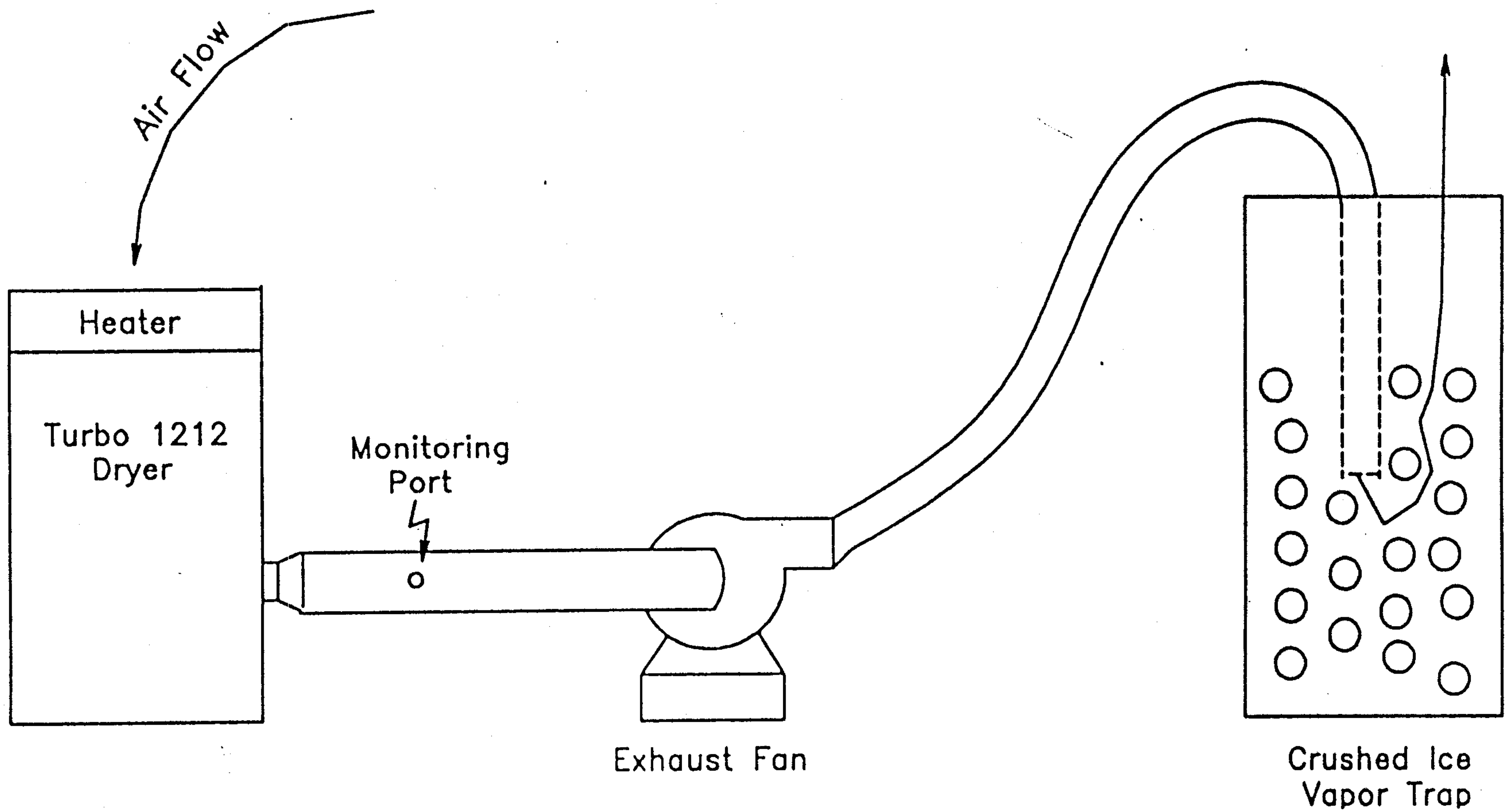
f). At the air exit end of the exhaust fan was attached a 6.0 ft. section of 6" diameter duct. This section of duct sloped upwards away from the exhaust fan at an angle of approx. 30 degrees for a distance of 4.0 feet.

The duct then was draped over the side and into a 25 gallon polyethylene drum*. (Hanging down into the drum approx. 2.0 feet.)

* The test was carried out in an area that had minimal ventillation of the air. In order to insure that the NMP vapors were kept in a confined area, and out of the general work area air, a vapor trap was constructed.

The 25 gallon polyethylene drum contained crushed ice cubes. The bottom 1.0 foot of the exit duct was submerged into the crushed ice cubes. This vapor trap was very effective at catching the NMP vapors that were exiting the testing unit. No NMP odor could be detected in the work area during the test. Throughout the test, as the ice would melt, the water was drawn off, and additional ice was added to the drum.

Figure 1: Modified Centrifugal Dryer Diagram



PART II: PREPARATION OF PARTS TO BE CLEANED

a). 37.98 lbs. of Hexagonal Cap Bolts (3/8" x 1 3/4") were initially added to the parts holding basket. These hexagonal bolts were left in the basket as the control weight for all the trials that were carried out.

b). (16) individual sets of (10) odd shaped metal parts (different shape and size bolts and nuts, than the hexagonal control bolts) were cleaned with acetone, allowed to dry at room temperature for two hours , and then each set was weighed to the 0.0001 gram.

c). Each set was then dipped into a room temperature bath of NMP; removed from the bath, and then allowed to sit on paper towelling for 20 minutes to wick off excess amout of liquid. Each set was then weighed to the 0.0001 gram, and then placed into a polyethylene jar. Dry nitrogen was used to blacket each jar prior to capping.

PART III: EXPERIMENTAL PROCEDURE

a). The dryer and exhaust fan* were turned on as soon as the control hexagonal bolts had settled up against the side of the spinning basket, the heater unit was turned on.

* Original plans called for running some experiments at different air flow rates, as the exhaust fan was set up with a control device to do this. However, with the inclusion of the ice vapor trap, the fan was able to maintain a flow rate (greatest) of 200 ft/ min.±5 ft./min.

b). The variable temperature control (digital read out) was initially set at 200ºF, and the control bolts/basket, were allowed to come up to temperature, as the temperature on the read out increased to the set reading.

Gradually, during the course of the trial run, the variable temperature control was changed to different set temperatures. Each trial, the bolts/basket were spinning, and changing temperature as the temperature of the unit regulated to the pre-set temperature for that particular trial.

c). A beaker of NMP was maintained at 140ºF, and as soon as the desired temperature in the dryer was attained, 35.0 - 40.0 grams of this hot NMP was added to the polyethylene jar containing the set of pre-weighed parts, for that particular trial.

d). After the desired temperature was attained, for each trial, the heater and dryer were turned off, and the top of the dryer was opened.

The contents of the polyethylene jar, (parts plus NMP), were poured into the dryer. The top of the dryer was closed, and the heater and dryer were turned back on.

e). The heater, dryer, and exhaust unit were allowed to run for the specified time for each trial. The heater and dryer were then turned off and the top of the dryer was opened.

f). Using a set of stainless steel tongs, the (10) control parts were removed from the dryer and set aside to cool down to room temperature.

g). A final weighing, of the set of (10) control parts, to the 0.0001 gram, was carried out to determine the amount of NMP removed and degree of dryness. (see figure 2)

h). The time that the dryer was run for each trial, as well as the temperatures recorded from both the thermocouple 1.5 inches below the heater coil, and through the port hole in the exhaust duct 0.5 ft. from the dryer exit port, are recorded in Figure 3.

PART IV: OBSERVATIONS AND CONCLUSIONS

OBSERVATIONS:

a). The average amount of NMP removed for the (6) trials run with the air input temperatures of 283ºF-293ºF (air exit temperature of 130ºF - 134ºF) was 99.71%

RANGE: 99.10% - 100.00%

b). The average amount of NMP removed for the (7) trials run with the air input temperatures of 230 - 250ºF (air exit temperature of 120º - 122ºF) was 97.46%

RANGE: 94.9% - 100.00%

c). Two trials had exit temperature of approximately 120ºF, and showed 100% drying of the control parts. One of these (2) trials had an air input temperature of 250ºF, and the second trial, although only having an air input temperature of 230ºF, was run for 18 minutes.

The other (5) trials having air exit temperatures of approximetely 120ºF, had air input temperatures in the 220ºF range, and gave 95 - 97% NMP removal.

CONCLUSION:

With an air flow rate of 200 ft/minute pulling the NMP out of the dryer the following drying cycle parameters should be followed.

1). Temperature of the air entering the drying chamber should be greter than 250ºF (121ºC). Most desireable temperature 280ºF (138ºC)

2). Drying time 3 minutes minimum.

PART V: SAFETY IN THE WORKPLACE

When working with any solvent at elevated temperatures, safe work practices should be followed. These experiments were run under tightly controlled conditions. The air flow of 200 ft/min was well above that required to insure no build up of NMP vapors, in a confined space, to the lower explosion limit.

To be secure that the proper safety precautions are built into any dryer, we recommend that the designer and user of the dryer both consult the National Fire Code (NFPA Section 86) to insure that the particular oven or dryer in question has the proper mechanisms or controls to prevent any safety related problems from occuring, while the unit is in operation or at rest.

NMP is a solvent, and like most solvents, it will de-fat/de-oil skin. When using NMP make sure that proper resistant Butyl of Neoprene rubber gloves are worn. Also, as with all solvents, goggles should be worn to protect the eyes from accidental splashes.

FIGURE 2
DRYNESS OF SETS OF (10) CONTROL PARTS
(NMP REMOVED)

Trial	Initial Weight (g)	Amount of NMP on parts (g)	Final Weight (g)	Weight Change (g)	% Weight Change From Initial Wt.	% NMP Removed
1	134.5975	0.6874	134.6380	+0.0405	+5.89%	94.11%
2	133.7760	0.5481	133.7966	+0.0206	+3.8	96.20
3	136.5165	0.5765	136.5163	-0.0002	0.00	~100.00
4	131.8631	0.6109	131.8636	+0.0005	+0.08	99.92
5	132.8241	0.4556	132.8286	+0.0045	+0.9	99.10
6	132.9919	0.4847	132.9898	-0.0021	-0.4	~100.00
7	133.7556	0.4411	133.7543	-0.0013	-0.3	~100.00
8	124.7264	0.4379	124.7287	+0.0023	+0.52	99.48
9	132.3927	0.4168	132.3938	+0.0011	+0.26	99.74
10	132.7262	0.4741	132.7432	+0.0170	+3.6	96.40
11	131.4378	0.4016	131.4551	+0.0173	+4.3	95.70
12	128.7020	0.4055	128.6981	-0.0039	-0.96	~100.00
13	132.4216	0.4396	132.4489	+0.0273	+6.2	93.8
14	133.4558	0.4384	133.4660	+0.0102	+2.3	97.7
15	132.8825	0.4700	132.9069	+0.0244	+5.1	94.9
16	134.3257	0.4174	134.3363	+0.0106	+2.5	97.5

FIGURE 3

(AIR TEMPERATURES AND DRYER DWELL TIMES)

TRIAL	Dryer Time (minutes)	Air Temperature At Thermocouple (ºF)	Air Teperature At Exhaust Monitoring Port (ºF)
1	6 minutes	200ºF	103ºF
2	5 minutes	230ºF	112ºF
3	7 minutes	250ºF	122ºF
4	3 minutes	285ºF	134ºF
5	2 minutes	287ºF	133ºF
6	4 minutes	287ºF	134ºF
7	3 minutes	293ºF	134ºF
8	3 minutes	283ºF	130ºF
9	2 minutes	283ºF	130ºF
10	3 minutes	238ºF	122ºF
11	2 minutes	236º F	120ºF
12	18 minutes	230º F	120ºF
13	6 minutes	201º F	113ºF
14	5 minutes	220ºF	120ºF
15	2.5 minutes	220ºF	120ºF
16	7 minutes	220ºF	120ºF

APPENDIX 1:

ON THE DAY OF THE TEST:

Barometric Pressure:	760.23mm Hg
Room Air Temperature:	76º F (24.4ºC)
Relative Humidity:	47.1%
Dew Point:	43.9ºF (6.6ºC)
Air Flow Rate Through Dryer:	200 ft./min. (39.25cfm)
Air Temp. 2.0 inches above ice in VOC trap	46.5ºF (8.0ºC)

APPENDIX 2:

Throughout the duration of the drying trials, monitoring tests were carried out, in order to gather some base data on the efficiency of the "crude" crushed ice air scrubber.

NMP is totally water miscible, and water wash scrubbers should be efficient devices for removing air borne NMP from exhaust streams.

Three different air monitoring pumps were used.

1). One pump was placed so that the charcoal tubes were placed directly into the duct work approximately 6.0 inches from where the air exited the dryer.
2). A second pump was placed so that the air could be monitored at a point approx 6.0 inches above the ice in the VOC trap.
3). A third pump was placed so that the air could be monitored at a point approx. 16.0 inches above the ice in the VOC trap.

a). Pumps 1 and 2 were turned on only when drying trials were being run.
b). Pump 3 was left in the on position for the entire day that the trial was being carried out.

EFFICIENCY OF SCRUBBER

TRIAL	PUMP 1 NMP MEASURED (mg/m^3)	PUMP 2 NMP MEASURED (mg/m^3)	NMP REMOVED BY ICE TRAP (%)
3	2782	369	86.7%
4	3801	1712	54.9%*
5	4333	1498	65.4%
6	3646	551	84.9%
7	2877	977	66.0%
8	3108	626	80.0%
9	3512	1185	66.3%
10	3261	1908	41.5%*
11	2843	459	84.0%
12	3089	478	85.0%
13	2349	644	73.0
		AVERAGE EFFICIENCY: ALL TRIALS	**72.0%**

* Due to the lack of constant vigilance over the crushed ice vapor trap, when the ice would melt leaving larger holes for the condensate vapors to flow out of, the efficiency of the vapor trap decreased.

For all instances when fresh ice was added, the efficiency was increased back up to the 80 - 85% range. This gives a good indication that with a properly designed water scrubber, the amount of NMP leaving a manufacturing plant in the exhaust air can be kept at a very minimal amount.

PUMP 3: Was turned on for a total of 404 minutes, essentially representing a 7 hour work day exposure to a worker that would be breathing in the air 16.0 inches away from the ice trap.

This pump operated at a rate of 3.5 liters/minute and collected 84.2mg of NMP over the 404 minutes time span.

0.0842g NMP collected = 8.50 x 10 $^{-4}$ moles of NMP
99.0 g NMP per mole
assume 22.4 liters volume per mole of NMP (close to STP conditions 8ºC air~760 mm pressure)
22.4 liters per = 1.0 mole NMP
x 8.50 x 10 $^{-4}$ mole NMP
0.0190 liters of NMP gas

In 404 minutes time at 3.5 liter per minute
1414 liters of air passed through the charcoal, and in this 1414 liters of air was 0.0190 liters of NMP
0.0190 liters of NMP = 1414 liters of air
(x) liters NMP 1,000,000 liters of air

x= 13.44 liters of NMP gas per 1,000,000 liters of air
or 13.44 parts per million (13.44 ppm)

A man working 16.0 inches from the surface of this crude ice trap, would have been exposed, during his entire 7 hour shift, to air containing an average 13.44 ppm of airborne NMP.

FIGURE 4: DRYING TEMPERATURES