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DISTILLING NITROCELLULOSE CONTAMINATED WASTE SOLVENT - SAFELY

In order to reduce operating costs in industry, many companies recycle wash solvents on site, because of the high cost of disposal of hazardous waste and replacement solvent. On – site recycling is a great option to ensure top quality, full reusability and reduce EPA liabilities.

Many facilities use inks and coatings containing Nitrocellulose (NC), which require special considerations because of the potential of an exothermic reaction under certain conditions. This white paper discusses a safe and proven method of taking advantage of the cost savings of distillation, by using system features and operating procedures, which have been designed and developed through years of experience in this industry.

Properties of Nitrocellulose

Nitrocellulose is chemically an ester of Cellulose with Nitric Acid, and is commonly encountered in a variety of inks and coatings used throughout industry. The amount of Nitrocellulose used in a typical ink/paint system ranges from 2 to 30%, based on total solids content.

It is a flammable material that becomes unstable at elevated temperatures above 320° F (160°C), and under certain conditions may react and decompose exothermically, resulting in toxic fumes including oxides of nitrogen and carbon monoxide. As long as atmospheric oxygen is not introduced onto the material, decomposition will take place without ignition, but will continue to react and burn until the process is complete. If a reaction occurs, it is imperative that no one attempts to open the inspection hatch of the distillation chamber under any circumstances, and that the room be evacuated and vented until the reaction is over, and the room is safe to re-enter.

The conditions needed to initiate a Nitrocellulose reaction include:

a. An adequate amount of NC in the waste stream, which is normally the case in some industries.

b. The lack of liquid solvent in the dry waste stream ie. turning to solids.
c. Temperature. At no time should dry NC be overheated, or be in contact with any surface at unsafe elevated temperatures. Under actual operating conditions, a reaction could be started at temperatures as low as 230°F (110°C) under the right set of conditions. With operating temperatures under this limit, NC decomposition will not take place.

FACTORS IN CHOOSING A SAFE DISTILLATION SYSTEM

In extracting solvent from NC based inks and waste solvent, the objective would be to distill under a relatively low controllable temperature range, and ensure that all of the conditions of temperature and dryness needed to start a reaction are not present at the same time.

Operating temperatures are a variable in industry, as different inks and coatings are normally used and cleaned with a variety of solvents with a wide range of boiling points. Low boiling flammable solvents such as alcohols, Ethyl Acetate and MEK are commonly used. These have a boiling point of less than 200°F (93°C), while new, high boiling, low VOC alternative solvents, such as some N-Methyl Pyrrolidone (NMP) based products, can have boiling points in excess of 400°F (200°C). This wide range of operating temperatures requires a high degree of versatility in a distillation system design.

A distillation system should incorporate several interlocked features to provide a safe, economical and efficient system, ideally suited to the specific requirements of Nitrocellulose. Any system used for this type of application should be controlled by a Programmable Logic Controller (PLC) for optimal operation and safety, and should include the following features.

1. **Automatic vacuum system**. A vacuum system is recommended for most solvents, but is particularly necessary with high boiling solvents of above 230°F (110°C). A good system design, such as a high performance liquid ring vacuum pump, can be effectively used to reduce the boiling point of the solvent to operate within a safe zone. For high boiling solvents, the operating temperature can often be reduced by up to 100°F (38°C) or more. Not only does this allow for a safer operating environment, but it also increases the overall efficiency of the system by reducing the warm up and cooling cycles, and is less prone to other phenomena such as foaming and fouling.

2. **Steep wall cone design**. A steep cone design for the waste receptacle is highly desirable for optimal efficiency, safety and ease of use. As the rate of distillation is directly related to the heat transfer surface area between the heating media and the solvent, a steep cone, either immersed in a large oil bath or surrounded by a recirculating hot oil jacket, provides an ideal configuration for this type of application.

   Under normal operating conditions, the distillation chamber should automatically be kept full of solvent through an auto fill feature, and as the distillation process goes on, solids naturally tend to fall to the bottom of the cone and remain saturated with solvent. As the largest heat transfer surface area is toward the top of the cone, where most of the heat is required for distillation, the sludge concentrates at the bottom, where the small surface area provides less heat transfer, and consequently cooler temperatures, than in conventional flat or dished bottom designs.

Steep cones are less subject to hot spots, where a possible accumulation of dried NC residue may exceed safe temperatures and cause a decomposition reaction.

As an added bonus, a steep cone can be fitted with a large, free flowing discharge gate valve at the bottom, making the removal of sludge a very routine, safe and easy procedure.

3. **Scrapers.** The benefits of a steep cone design are enhanced by the use of self adjusting, low maintenance scrapers, which continually sweep the inside surfaces to help the solids to fall to the bottom, and prevent any build up of material, which could possibly result in an exothermic reaction. A scraper system should have a hub design which would not restrict a thickened sludge to flow freely during the discharging operation.

4. **Temperature control**. The principal area of concern in a safe distillation process is in controlling temperatures within the critical zone limit below 230°F (110°C) with precision and consistency. Au automatic solvent distillation system should monitor and control the temperatures in the sludge, vapor space and hot oil. By using Resistance Thermal Devices (RTD), accurate to within 1 degree, the process can be automatically controlled with accurate and consistent results.

   For Nitrocellulose applications, the most critical parameter lies in the sludge temperature. By monitoring and controlling this temperature precisely, the system will automatically prevent the sludge of ever reaching the reaction temperature. The system's PLC program should only allow the distillation process to operate within a specified range. This range should be protected by password security, to ensure against accidental changes by unauthorized personnel.

5. **Auto Cool**. Since a Nitrocellulose reaction could ignite in the presence of atmospheric oxygen, an auto cool system should be installed as part of the system. When the sludge temperature has reached the preset level, indicating that the system is ready to discharge, the PLC should automatically start an auto cool cycle, to rapidly cool the sludge to a safe discharge temperature, typically around 125°F (50°C). The sludge could then be safely discharged and handled in accordance with local safety practices and environmental regulations.

   Some systems offer a water quench option in the distillation system in the event of a reaction. Although most distillation systems are equipped with a pressure release valve on the distillation chamber, the introduction of water on an exothermic reaction may result in steam and pressure, and should be considered a last resort.

   In certain cases, where a distillation system is not equipped with the safety features required for this application, adding alkali or oil to the NC material may reduce the risk of decomposition. By adding caustic soda (NaOH) or soda ash (Na_2CO_3), a chemical process of the Nitrocellulose is started, which reduces the potential of decomposition and a reaction.

   The best approach to safety, however, lies selecting a system designed to prevent an accidental reaction in the first place.

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