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Assuring process safety in the transfer of hydrogen cyanide manufacturing technology☆

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Abstract

This paper outlines the critical issues to be addressed in the transfer of hydrogen cyanide (HCN) manufacturing technology to a licensee. Process safety management (PSM) is of critical importance because of the toxicity, flammability and reactivity of HCN. The critical issues are based on experience that DuPont has gained (1) while safely manufacturing hydrogen cyanide for over 50 years, and (2) while DuPont has safely licensed HCN technology to other firms at locations around the world. DuPont's HCN experience has been combined with Aker Kvaerner's project engineering experience to insure the safe transfer of HCN technology to a licensee.

Keywords: Hydrogen cyanide; Process safety management; Design; Hazards; Flammable

1. Introduction

The hazardous properties of hydrogen cyanide (HCN) require process safety management (PSM) to be the highest priority in design, engineering, construction, start-up and operation of a HCN plant. And these principles can also be applied to a variety of hazardous and toxic materials such as phosgene, chlorine, oleum and other chemicals. Ensuring that safety related information is properly communicated from the technology licensor to the engineering contractor, to the operating staff of the licensee, and to the project decision makers is critical to ensuring the construction of a safe plant, regardless of the product it makes.

DuPont has manufactured HCN safely for over 50 years. During this period DuPont has also transferred their HCN manufacturing technology safely to several other companies.

The Engineering and Construction (E&C) business area of Aker Kvaerner specializes in providing process technology, design, engineering, project management, procurement and construction services. Aker Kvaerner safely licenses and designs a wide range of process technologies for the petrochemical, chemical and polymer industries. Aker Kvaerner E&C has a leadership record with regard to project safety.

In 2000 Aker Kvaerner and DuPont partnered on one of DuPont's proprietary hydrogen cyanide (HCN) process technologies. One of the goals of this partnership is to insure that future HCN plants are built and operated safely—because severe and well-publicized HCN incidents could have repercussions for all HCN producers and consumers.

2. Hydrogen cyanide information

Hydrogen cyanide (chemical formula: HCN) is a chemical reagent used in the manufacture of a wide range of industrial products as shown in Table 1.

Hydrogen cyanide is a colorless gas or low viscosity liquid (normal boiling point is 26 °C) with a bitter almond odor. It has the following hazardous characteristics:

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Table 1
Products produced from hydrogen cyanide

Chemical	Application
Methyl methacrylate	Coatings and acrylic sheeting
Adiponitrile	Nylon production
Sodium cyanide	Precious metals extraction
Methionine	Animal feed supplement
Ibuprofen	Pharmaceutical
Glyphosate	Agricultural chemicals/herbicides

Table 2
Toxicity of hydrogen cyanide

HCN concentration (ppm)	Consequence
2–5	Odor threshold
10	Exposure limit (PEL)
20–40	Slight symptoms after several hours
45–54	Tolerated for 30–60 min without significant immediate or delayed effects
100–200	Fatal after 30–60 min
300	Immediately fatal (without first aid)

Note. The United States OSHA permissible exposure limit (PEL) is 10 ppm while some states have an exposure limit of 4.7 ppm. Other countries may have exposure limits that are different from the United States limits.

Toxicity. Fast-acting and highly poisonous material as illustrated in Table 2.

Flammability. Highly flammable with a National Fire Protection Association (NFPA) flammability rating of 4 (severe fire hazard). This can be an advantage in some cases because HCN spills or releases can be safely burned to less toxic combustion products.

Reactivity. Tendency to undergo exothermic runaway reactions that cannot be vented. Addition of alkaline chemicals such as NaOH, amines or NH_3 must be avoided since they promote polymerization and decomposition of HCN. These are exothermic reactions that can result in a rapid increase in temperature and pressure, and may lead to loss of containment. Small quantities of acid (sulfuric, phosphoric and acetic) are used as a stabilizer for liquid HCN to prevent decomposition reactions from occurring. Small quantities of sulfur dioxide are sometimes used to stabilize HCN vapor that may condense in tanks and vessels. Addition of large quantities of sulfuric acid (HCN/H_2SO_4 mole ratio of 0.5–1.0) can also cause HCN decomposition.

Table 3
Compare HCN hazards to hazards of other chemicals in the HCN process

	U.S. RQ (lb)	U.S. PEL (ppm)	NFPA ratings		
			Flammability	Reactivity	Health
HCN	10	10	4	2	4
Ammonia	100	50.0	1	0	3
Natural gas	None	None	4	0	1
Sulfuric acid	1000	1.0	0	2	3
Sulfur dioxide	1	5.0	0	0	2

Polymerization. Tendency to form solid polymerization products which can plug relief systems, instrumentation, valves and piping, thereby causing or exacerbating process hazards.

Hydrogen cyanide is listed in the United States OSHA regulations (29 CFR Part 1910.119, Appendix A), "List of Highly Hazardous Chemicals, Toxics and Reactives" with a threshold quantity of 1000 pounds. The reportable quantity (RQ) for releases is 10 pounds.

Although HCN is the most toxic material in the Andrussow HCN process, other chemicals can also be hazardous—as illustrated in Table 3.

3. DuPont HCN technology

DuPont and Aker Kvaerner license the modified Andrussow process for HCN production. In this process oxygen from air, ammonia and natural gas react in a gas phase converter to produce hydrogen cyanide, water and combustion products as shown in Fig. 1.

The HCN reaction is shown below:

$$NH_3 + CH_4 + 1.5O_2 \xrightarrow[\text{Pt/Rh}]{1200\,^{\circ}\text{C}} HCN + 3H_2O,$$

$$\Delta H = -482.3\,\text{kcal/mol HCN}$$

Unreacted ammonia is recovered from the gas that leaves the converter and is recycled back to the HCN converter. The unreacted ammonia must be removed from the converter discharge gas to ensure that acidic conditions are maintained in the HCN refining train to avoid the formation of HCN polymer. An acidic environment is maintained by acid addition in the HCN refining train. An acidic environment is needed to stabilize the HCN. The crude HCN product is then distilled to high purity (>99.5%) in

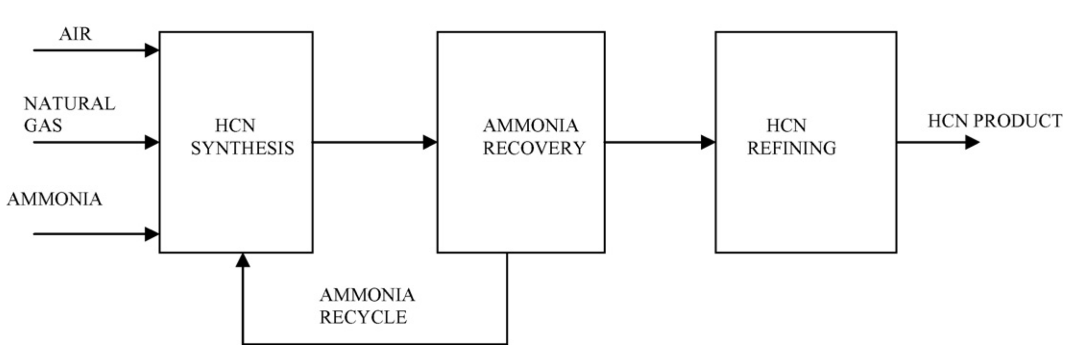

Fig. 1. Block diagram of DuPont/Aker Kvaerner HCN process.

the HCN refining train. The exothermic heat of reaction in the converter is used to produce steam in a waste heat boiler.

4. Process safety management in plant design

The approach that should be used by any company considering construction of a chemical plant that produces or handles a hazardous chemical should be to:

- First, gain a complete understanding of the hazards inherent in the substances being handled.
- Then, as early as possible in the project design phase, evaluate inherently safer plant design alternatives.

Throughout the life cycle of the project, formal safety reviews should be conducted including:

- process design safety review,
- appropriate level of process safety analysis, and
- pre start-up safety review.

[see OSHA regulations for highly hazardous chemicals—29CFR 1910.119 (i)].

The appropriate level of process safety analysis is very important to consider. For example, some companies that the authors have visited may only perform one type of safety analysis—no matter how complicated the process is and how limited their experience is with the process. However the Andrussow HCN process has some steps that should lead a first-time operator to conduct a fault tree analysis—such as the converter operation. This is because of the complex control system that is needed to maintain safe conditions during start-up, normal operation and shutdown. On the other hand, the boiler feed water system for the waste heat boiler on the HCN converter could have a safety analysis based on a checklist or "What-If" procedure.

5. Hazards inherent in the substances handled

When the process technology to be used in a new plant or in the retrofit of an existing plant is licensed, the licensee should make sure that the licensor is involved at critical stages of the project. Because of the hazardous nature of the chemicals being handled, the licensee needs the assistance and guidance of qualified personnel from the licensor to ensure the safe transfer of the selected technology. During the evaluation of technology licensors, the licensee should make sure that the selected technology licensor is able to provide the required project support during all phases of the project.

To ensure that the licensed process technology is operated safely, it is critical for all parties (licensor, the engineering contractor and the licensee's operating personnel) to understand the inherent hazards associated with the chemicals being handled. In general, there is no substitute for plant operating experience when it comes to understanding how hazardous chemicals should be handled. For this reason a licensor who operates plants using the technology to be licensed will be able to impart a better understanding of these hazards to the licensee.

There are hazards inherent in the substances handled in a HCN plant, as clearly illustrated in Section 2. DuPont's unique experience in safely operating HCN plants for over 50 years provides the basis for their understanding of these hazards.

For plants producing hazardous chemicals, it is more critical for the licensee to involve the potential licensor in the initial project planning even if this interaction occurs prior to a license agreement. At this very early stage of project development the licensor can provide valuable input into the following project activities:

- hazards identification,
- lines of defense against hazards,
- plant siting and layout studies,
- consequence and risk assessment studies,
- audits of existing facilities (particularly emergency response capabilities),
- analysis of human factors that could affect the project,
- assessment of storage requirements,
- evaluation of alternatives,
- communication of design strategies.

A key to having a complete understanding of the inherent hazards associated with the chemicals is having information on the chemical incompatibilities that may exist in a facility. This information is best presented in a chemical interaction matrix and should include feedstocks, intermediates, chemicals, products, utilities and materials of construction. The chemical interaction matrix is a very efficient means of ensuring that this specific knowledge is transferred from the licensor to all other participants in a project. It can provide unambiguous guidance for the licensee and engineering contractor during the detailed engineering phase of the project. Part of a chemical matrix for HCN is shown in Fig. 2:

Some specific hazards associated with the production of HCN and the resultant control, protection and mitigation measures employed by DuPont are discussed below.

Based on a study by Fauske Associates (that was supported by a consortium of HCN producers and consumers), HCN vents cannot be sized to relieve the overpressure due to runaway reaction/polymerization scenarios. Therefore to prevent HCN polymerization and decomposition reactions from occurring in the HCN refining train, the following steps have been taken to maintain a safe operation:

- Dilute acid sprays are located in strategic locations in equipment, vent lines, pressure relief valve inlets and other stagnant flow locations;
- Sulfur dioxide (SO_2) is used as a vapor phase stabilizer in the HCN refining train;
- Special operating techniques are used;
- Special design procedures are employed;
- Process protection (interlocks and independent process protection devices) is provided.

The feedstocks to the HCN converter can form flammable mixtures that could explode in the presence of the hot (1200 °C)

		1 carbon steel	2 stainless steel	3 hastelloy C276	4 alloy 20	5 incoloy 800	6 inconel 601	7 stainless/graphite gaskets	8 glass filled teflon gasket	9 barium sulfate filled teflon gasket	10 Turbine oil	11 Zinc Phosphate	12 Scale Inhibitor	13 citric acid	14 glycolic acid	15 sulfamic acid	16 Nomex ®	17 Polypropylene	18 Cotton
1	acetonitrile	n	n	n	n	n	n	n	n	n	n	rh	rg	rh	rh	rh, rg	n	n	n
2	acrylonitrile	n	n	n	n	n	n	n	n	n	n	rh	rg, p	rh, p	rh, p	rh, rg, p	n	n	n
3	Air	n	n	n	n	n	n	n	n	n	n	n	n	n	n	n	n	n	f
4	Alumina	n	n	n	n	n	n	n	n	n	n	n	n	n	n	n	n	n	n
5	ammonia	n	n	n	n	n	n	n	n	n	n	rh	n	rh	rh	rh	n	n	n
6	ammonium formate	n	n	n	n	n	n	n	n	n	n	n	rh	rh	rh	rh	n	n	n
7	ammonium phosphate	n	n	n	n	n	n	n	n	n	n	n	rh	rh	rh	rh	n	n	n
8	Antifoam (silicone, methylcellulose)	n	n	n	n	n	n	n	n	n	n	n	n	rh	rh	rh	n	n	n
9	carbon dioxide	n	n	n	n	n	n	n	m	m	n	n	n	n	n	n	n	m	n
10	carbon monoxide	n	n	n	n	n	n	n	n	n	n	rh	rh	rh, e	rh	rh	n	n	n
11	caustic (NaOH)	n	n	n	n	n	n	n	n	n	n	rh	n	rh	rh	rh	n	n	n
12	fiberfrax	n	n	n	n	n	n	n	n	n	n	n	n	n	n	n	n	n	n
13	Freon 22	n	n	n	n	n	n	n	n	n	n	n	n	n	n	rh	n	n	n
14	HCN	n	n	n	n	n	n	n	s	s	n	n	p	n	n	n	n	n	n
15	HCN / Hydrogen Mixture	n	n	n	n	n	n	n	n	n	n	n	rh	rh, rg	rh	rh, rg	n	n	n
16	Layonite (limestone)	n	n	n	n	n	n	n	n	n	n	rh	n	rh	rh	rh	n	n	n
17	methanol	n	n	n	n	n	n	n	n	n	n	rh	rh	rh	rh	rh	n	n	n
18	natural gas	n	n	n	n	n	n	n	n	n	n	n	n	n	n	n	n	n	n
19	phosphoric acid	n	n	n	n	n	n	n	n	n	n	n	rh	rh	rh	rh	n	n	n

rh = reaction with heat
f = flammable
rg = reaction to release toxic gas
ro = oxidizing reaction
p = polymerization, with heat and pressure
m = melt
e = explosive
s = swells
n = no hazard

Fig. 2. Excerpt from chemical interaction matrix for HCN process.

catalyst. To avoid the formation of a flammable mixture, the Memphis HCN plant:

- Uses extensive process controls and operator training to keep the mixture of air, ammonia and natural gas that is fed to the HCN converter outside the flammable composition range;
- Has designed equipment and relief systems to protect equipment and personnel from the potentially adverse consequences of a deflagration.

[*Note*. Deflagration is a process of subsonic combustion that usually propagates through thermoconductivity (i.e., hot burning material heats the next layer of cold material and ignites it). Deflagration is different from detonation, which is supersonic and propagates through shock compression.]

The HCN process hazards are mitigated by providing multiple, independent levels of protection. These levels of protection are provided by:

- rigorous operator training and procedures,
- careful design and calibration of the instrumented control system and its algorithms,
- redundant instruments—where appropriate,
- safety alarms and interlocks, and
- adequately designed overpressure protection—based on the guidelines in NFPA 68 (Guide for Venting of Deflagrations).

As well as considering deflagrations in vent design, DuPont has developed design criteria to ensure that a transition to a detonation does not occur in the HCN converter system. These criteria include the following:

- frequent calibration of air, ammonia and natural gas flowmeters,
- frequent checks on interlocks related to the converter feed flowmeters,
- establish a minimum operating rate to insure that orifice and venturi flowmeters provide accurate flow readings,
- frequent checks on the interlocks on the converter reaction temperature and
- avoiding the intrusion of other gases (such as nitrogen, carbon dioxide, steam, etc.) into the natural gas and ammonia so that the true flow rate of the reactants is being measured.

6. Inherently safer plant design

Elimination of process hazards by inherently safer plant design is preferred over protection or mitigation measures. The design features of an inherently safer plant design will often fall outside of normal design parameters (e.g., equipment design pressures may be higher than would be expected based on operating pressure only).

During technology selection, the potential licensee has the opportunity to make a big impact on the safety performance of the final plant. Technologies should be evaluated with a strong preference for plant designs that incorporate inherently safer design features. The best way to accomplish this evaluation is to ask the licensor to clearly describe the inherently safer design features in their technology offering—because they would be more qualified to recognize opportunities and the pitfalls of different designs.

Some of the topics that should be discussed as a part of the inherently safer design evaluation are:

(1) intensification (or minimization)—by using small quantities of hazardous materials,
    (a) for example, make and immediately consume a toxic intermediate to limit the quantity in the process to a minimal amount.
(2) substitution/elimination—by replacing hazardous materials with less hazardous ones,
(3) attenuation (or moderation)—by using a less hazardous form of a material or by using less hazardous process conditions (such as a pressure closer to atmospheric or a temperature closer to ambient),
(4) limitation of effects—by designing the facilities to minimize the impact of a release of a hazardous material,
    (a) for example, build process vessels strong enough to withstand the highest pressure that could be generated inside the vessel. This would eliminate the possibility of failure due to over-pressurization and the need for pressure-relief devices.
(5) simplification/error tolerance—by designing the facilities so that operating errors are less likely and facilities are forgiving when errors do occur.

The inherently safer design is best achieved (1) by the licensor including this topic in the written discussion on safety in the technology license package and (2) by the licensors' attendance at project safety reviews that include the appropriate level of safety analysis [such as checklist, hazard and operability study (HAZOP), fault tree analysis (FTA) and/or failure mode and effect analysis (FMEA)].

The DuPont HCN process technology makes extensive use of inherently safer design features to mitigate hazards. Some of these design features include:

- minimization of pumps in the HCN refining train,
- minimization of HCN inventories in HCN refining train column reflux/overhead equipment,
- relief valve set pressures ensure liquid boiling during an exothermic polymerization reaction assists in absorbing heat generated without rapid temperature increase,
- plant layout to avoid adjacent storage of incompatible chemicals,
    - ○ field fabrication of piping without flanges—in certain services,
    - ○ use of proven materials of construction—including gaskets,
    - ○ use of safe and efficient vessel isolation equipment that avoids swinging blanks, and
- in case of a plant upset or emergency, the process contents can be safely directed to a dedicated system that either:
    (a) disposes of the process contents in a controlled manner or
    (b) safely vents the process contents to avoid equipment damage.

7. Safety during development of the license package

Plant safety can best be assured by the licensor gaining an understanding of each licensee's unique requirements. Some of the items that should be discussed include:

- Understand the downstream processing requirements:
    - o minimizing the inventory of liquid HCN product or other hazardous chemicals is a desirable goal, and
    - o under certain circumstances HCN liquid storage tanks can be eliminated by careful HCN plant control system design.
- Vessel entry and equipment cleanout procedures are affected by:
    - o the licensee's maintenance and operating procedures,
    - o the piping and equipment that is provided for these activities, and
    - o facilities available to process and treat residual contents in vessels.
- Consequence and risk analysis, including plume modeling of vapor release scenarios, may dictate:
    - o where the plant is located and
    - o whether additional protective measures will be required.

To ensure that a technology package includes all site-specific conditions, the licensor should visit the licensee's facility and evaluate the following items:

- training and experience of the operating personnel,
- site layout and prevailing wind direction,
- locally applicable regulations, codes and standards,
- site risk management plans and emergency preparedness,
- process control systems,
- operating and maintenance philosophies,
- feedstock, product and intermediate storage requirements,
- piping standards,
- fire water supply availability, and
- management of change procedures during design, construction and operation,

A plant visit by the licensee to an operating plant using the licensed technology will usually occur after the license agreement has been finalized. As well as being critical for the licensee to familiarize themselves with and evaluate the process technology, this is also a unique opportunity for the licensor to highlight safety issues and begin the transfer of important safety information to the licensee's organization.

DuPont and Aker Kvaerner have conducted process design safety reviews on HCN facilities. These reviews utilize a "What

If/Checklist" technique to identify safety concerns and issues. This checklist is used in the preparation of a technology license package to ensure that safety issues are adequately addressed in the package and communicated efficiently to the engineering contractor.

Communication of safety related issues to the engineering contractor is greatly enhanced by ensuring that the technology license package has adequately documented the safety considerations. This is achieved by including the following documents in every technology license package:

- control philosophy—including standard operating conditions and alarm settings,
- trip and interlock schedules—including settings,
- a substantial written discussion on safety issues and safe practices,
- a product and service index—which describes the piping materials for different chemical and utility services,
- process design safety review report—including a list of follow-up actions for the detailed engineering phase,
- relief load summary—including vent sizing for potential deflagrations where applicable,
- preliminary plot plan layout,
- sample point design for protection of personnel,
- safety and fire protection review—including initial specifications for gas detection and fire suppression systems.

8. Safety during detailed engineering

In every project the engineering contractor and the owner will have varied experience and this will determine the amount of time the licensor devotes to detailed engineering activities. To ensure the highest level of safety, the licensor should be involved in specific detailed engineering activities for plants producing highly hazardous chemicals.

Once detailed engineering has commenced, consultation by the licensor on the following safety related issues would be highly recommended:

- plant layout,
- model reviews,
- safety reviews,
- pump mechanical seal selection,
- preparation of start-up manuals,
- preparation of operating manuals,
- preparation of technology manuals,
- design review of safety critical systems, and
- design review of critical equipment.

Specifically for DuPont's HCN technology, the following detailed engineering activities should be reviewed by the licensor:

- piping design in critical areas to ensure no stagnant zones exist where HCN polymerization reactions can occur,
- equipment design that will minimize the potential for polymerization,
- equipment design that will maximize process uptime—which will help minimize the potential for exposures to hazardous conditions,
- locations of fixed point HCN gas detectors,
- locations and arrangements for acid stabilization sprays and inhibitor addition,
- design for critical safety systems including instrumentation and controls to prevent flammable gas mixtures in the feed to the HCN converter,
- location of safety showers and eye wash stations,
- location of oxygen, cyanide antidotes and fire blankets, and
- sampling point details and locations—because fatalities have occurred during HCN sampling at other companies.

9. Training, commissioning and start-up

Because of the toxicity and reactive properties of HCN, it is critical that specific operator and mechanic safety training be conducted. This training will ensure that all personnel are fully aware of the safety issues, the recommended safe practices, and are able to react appropriately to emergency situations. The training for HCN and other hazardous chemicals should include topics that should be presented by the technology licensor. For DuPont's HCN technology these topics may include:

- how to prevent polymerization reactions,
- hazardous properties of HCN,
- hazardous properties of other raw materials (natural gas, ammonia, and acids that inhibit polymerization),
- odor detection training for HCN,
- recognizing symptoms of HCN exposure,
- first aid and rescue procedures,
- personal protective equipment use such as:
  - the use of portable, electronic, individual HCN detectors,
  - continuous communication (via portable radios) between control room and field operating personnel and
  - the wearing of fire-resistant clothing while working in the HCN plant.
- normal operating procedures,
- how to respond to process upset conditions,
- equipment isolation for maintenance work,
- equipment decontamination procedures,
- maintenance procedures—including "first breaks",
- a schedule for follow-up training of personnel to insure that they maintain their skills,
- a schedule for reviews of operating and maintenance procedures, and
- a schedule for process hazards analysis of different sections of the process to insure continuing improvement in the safety of the operation.

DuPont has many years of experience with the safe operation of HCN plants and their experienced operating and maintenance personnel would be utilized in any personnel training. Other companies that license technology for the production of hazardous chemicals should also be expected to use experienced personnel to train the licensee's operating and maintenance personnel.

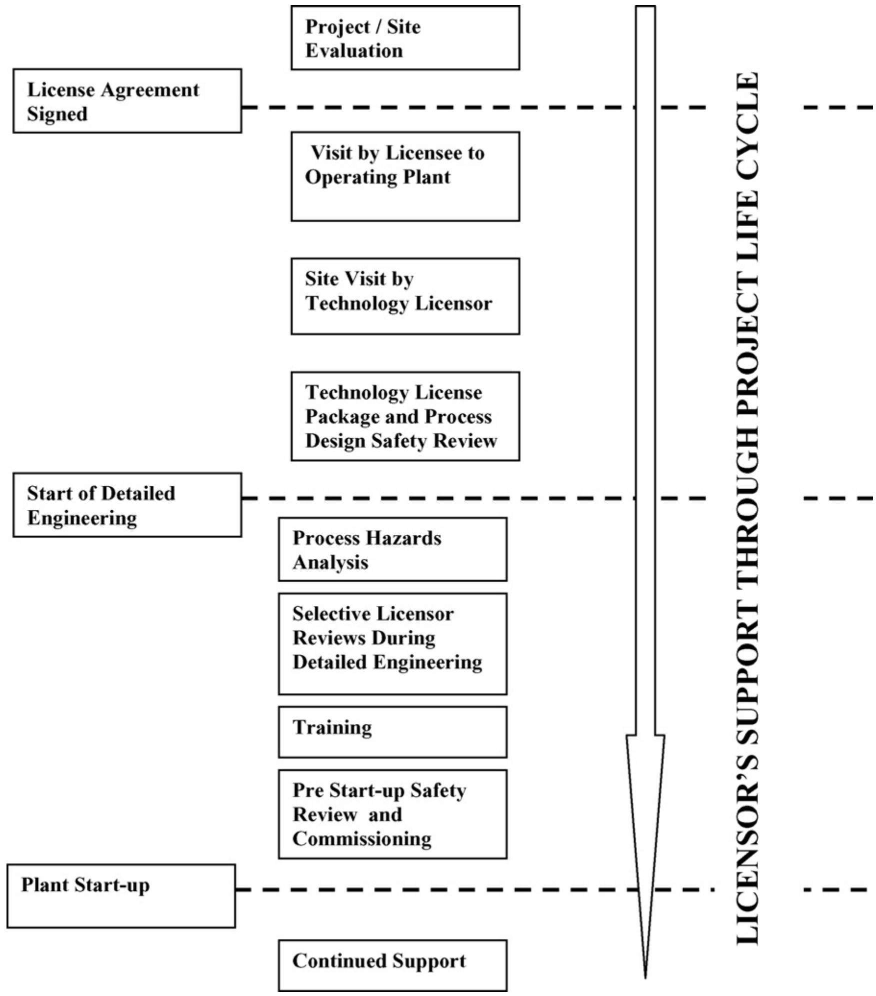

Fig. 3. Licensor activities to ensure the safe transfer of licensed process technology.

Commissioning is one of the key steps in achieving a successful and on-time start-up. DuPont and Aker Kvaerner have extensive experience with:

- vessel inspections,
- appropriate pipe cleaning procedures,
- removal of contaminants that can reduce operating efficiencies,
- instrument check-out procedures,
- process controls simulation,
- water runs and other dummy tests to check-out the operation prior to start-up,
- preparation of a start-up manuals, and
- other commissioning activities.

The technology licensor must be present for the plant start-up, and it is strongly recommended that these personnel take part in a formal pre-start-up review. This review will normally be conducted just prior to the beginning of the start-up activities. This review should verify that:

- proper training has been provided,
- operating procedures are complete,
- emergency systems are operational,
- critical instruments are calibrated correctly, and
- the emergency response plan and equipment are in place.

Once a plant has been started up and lined out, the process technology licensor can provide continued support in a number of safety related areas:

- auditing and benchmarking,
- review of process improvements,
- review of capacity expansion plans,
- continued training,
- incident investigations,
- consultation on specific safety issues,
- advice on technology improvements, and
- involvement in periodic revalidations of previous process hazard analyses.

10. Conclusions

HCN process technology and technology for other hazardous chemicals can be safely transferred to licensees. As illustrated in Fig. 3, this transfer can be successfully accomplished by communicating and implementing process safety management (PSM) elements during:

- technology transfer,
- design,
- construction,
- commissioning,
- start-up, and
- routine operation.

Engineering, procurement and construction (EPC) of process plants that use licensed process technology presents many opportunities for failure to communicate and implement key PSM elements. One of the reasons for the failures is that different organizations may be responsible for technology transfer, design, construction, commissioning, start-up and operation. These problems can be mitigated by selecting a process technology licensor who, as well as providing process technology, is committed to providing guidance and expertise on safety issues throughout the project life cycle. When considering the licensing of process technology for a hazardous product – such as hydrogen cyanide – the choice of a licensor with both operating and licensing experience should be paramount in the licensee's decision making process.

Hydrogen cyanide is a useful chemical reagent for the manufacture of a wide range of valuable industrial products. Hydrogen cyanide's physical, chemical, and toxicological properties present significant process hazards in its manufacture and use. Despite these challenges, for over 50 years DuPont has safely manufactured hydrogen cyanide and licensed the Andrussow HCN technology.

The DuPont/Aker Kvaerner alliance utilizes their experience and safety awareness to insure the safe transfer of HCN manufacturing technology. This type of alliance between a producer of hazardous chemicals and an EPC company could be used as a model for the safe transfer of other hazardous chemical technologies.