

LOUISIANA DIVISION
CONSOLIDATED AUDIT

Reactive

Chemicals

MARCH 1991

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Dow U.S.A.

The Dow Chemical Company
P.O. Box 150
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Louisiana Division

LOUISIANA DIVISION GUIDELINES FOR REACTIVE CHEMICALS REVIEW

The enclosed package contains guidelines to assist you in preparing for a productive and comprehensive Reactive Chemicals Review. It is important to realize that Reactive Chemicals considerations are YOUR responsibility. The Reactive Chemicals Committee is made up of people experienced in this area and helping you fulfill your reactive chemicals responsibility is our primary goal.

Evaluation of past incidents shows that inadequate operating procedures, lack of positive raw material identification, critical instrument problems, and material of construction incompatibility cause many problems. Also, waste handling and water contamination continue to be serious concerns. Consider these items carefully.

Attached is a Reactive Chemicals Review checklist which will guide you in preparing for your review. Please complete this checklist and include as the cover sheet for your review package. Attachments are also provided for updating Reactive Chemicals data and to assist you in developing "Worst Case" scenarios. A list of resources to help you prepare is included.

The final attachment is a self evaluation questionnaire which we encourage you to fill out to help you recognize strengths and weaknesses of your program. We think this questionnaire will be an asset to you in further development of your Reactive Chemicals Program. The results of the self evaluation should be a topic of discussion at your review.

Make every attempt to limit your review presentation to chemical reactivity. The Reactive Chemicals Committee does not deal with other problems unless they bear on chemical hazards. Do not review markets, costs, strategy, etc.

The Plant should send one copy of the completed Questionnaire to the LAD Audit Facilitator 10 calendar days before the audit. Send 10 copies of the presentation packet to Cindy Gum (Reactive Chemicals Office Professional, Building 2510, Ext. 1601) at least 1 week prior to the review.

Gerald Wagener, Chairman
Reactive Chemicals Committee
Building 2511, Ext. 1828

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REACTIVE CHEMICAL REVIEW CHECKLIST

PROCESS _____
F LDING _____

REVIEW DATE _____
PRESENTER _____

ADMINISTRATIVE

- _____ Review scheduled through Cindy Gum, 389-1601
- _____ Mini or Full review (Circle one)
- _____ Type of review - New Super., 2 year, Process change, Project (Circle one)
- _____ 10 copies of Review package (copy of slides) sent to Cindy Gum 7 days prior to review
- _____ Safety Superintendent invited to review
- _____ R&D contact, process engineer, Tech Center contact etc., invited to review
- _____ Appropriate supervision invited to review

DATA

- _____ Appropriate interpretation of data _____ Physical Properties
- _____ Screening tests complete _____ Literature data
- _____ Reactive Chem Global database searched _____ Unusual properties
- _____ Calculated thermodynamic data

CHEMISTRY

- _____ Equations for each reaction (side and main)
- _____ Comparison of Calculated vs. Measured heats of reaction
- _____ Reactivity matrix (inter-reactivity of all process materials)
- _____ Impurities

SYSTEM

- _____ Pressure relief system designed _____ Critical instruments
- _____ Process conditions (temp, press.) _____ Materials of Construction
- _____ Quantities _____ Equipment size

MISC.

- _____ Recommendation from last review _____ Tech Center input
- _____ History of related R.C. incidents _____ F&E Index complete
- _____ Waste storage and disposal _____ Training activities
- _____ Worst case scenarios/lines of defense determined
- _____ Discussions with Safety Superintendent, R.C. Personnel or other resources

PACKAGE COMPLETENESS

- _____ Statement of purpose
- _____ Summary of major reactive chemicals concerns
- _____ Process chemistry--include kinetic and thermodynamic data (Measured vs. calculated)
- _____ Physical property summary for all chemicals handled (Boiling point, vapor pressure re, flammable limits, flash point, auto ignition temperature, Materials Handling- Include how the Plant receives, identifies, stores handles and disposes of materials.
- _____ Simplified Process Flow Sheet - include control schemes, monitoring and relief devices, points where there is reactive chem data, equipment size and location, process conditions and quantities.
- _____ Summary table of Reactive Chemical data - include raw materials, intermediates, products, by-products and wastes.
- _____ Reactive Chemical concerns for each step - include concerns for each step in the process and worst case scenarios and calculations. Cover the steps taken to prevent an incident, or lines of defense. (see attachment A)
- _____ One copy of actual Reactive Chemicals Report (i.e. ARC traces) at meeting.

GUIDELINES FOR UPDATING REACTIVE CHEMICAL DATA

Briefly review each data sheet/report for the following:

1. Are the materials relevant to and involved with the current process?
(Yes - proceed, No - discard)
2. Is the test data later than 1-1-82? (Yes - put in "Active" Data file/book, if not - put in "Review" stack)
3. Make two lists of materials tested: One will be the "Active" file and the other the "Review" file. Include the data sheet number, date, and the results. Take the lists to the Reactive Chemicals Testing Lab, R&D Bldg. 2511 with a brief verbal or written description of the process, simplified index flowsheets, volume of the materials and what potential conditions can occur. Advise if more extensive testing will be available.

THE "WORST CASE" TEST

The owner of an operation with reactive chemicals hazard potential should make of the "Worst Case" thinking in evaluating the safety of his operation. At every point in the operation where an exothermic reaction may take place, the owner should conceive the worst possible combination of conditions which could exist. This may include such conditions as loss of cooling water, wrong combination of reactants, effect of contaminants, materials of construction, closed valves, plugged lines, instrument failure, air leakage, loss of agitation, deadheaded pump, mishandled catalyst, or combinations of above. An engineering evaluation should then be made of the consequences of the "Worst Case" conditions.

Potentially serious consequences of a "Worst Case" situation may rule out practicing the operation. Usually, adequate means of avoiding "Worst Case" conditions and coping with the consequences can be devised. Where serious "Worst Case" consequences are recognized, the owner must identify and implement adequate lines of defense. These should start with preventive measures, followed by corrective measures, then containment measures, and, as a last resort, isolation techniques.

Redundancy is essential for protection against reactive chemicals accidents. The owner should consult with the Reactive Chemicals Committee and his supervisor on the adequacy of his lines of defense.

Unreliability of protection systems must not be allowed to be a cause of an accident. Regular documented testing of all elements of protection systems is essential. This testing includes relief devices, critical instruments, and operator knowledge.

HAZARDOUS OPERATIONS - LINES OF DEFENSE

- First Line Knowledge of Reactions, R.C Screening, Good Process Design, Proper Operating Instructions, Alert Operation.
- Second Line Warning Devices, Temperature and Pressure Cut-Offs. Feed Cut-Offs, Level Alarms, Etc.
- Third Line Relief Valves, Vents, Deluge Systems, Etc.
- Fourth Line Appropriate Isolation.

RESOURCES TO HELP YOU PREPARE

1. Louisiana Division Reactive Chemicals Program Manual
2. Existing reactive chemicals data from plant files or the Global Reactive Chemicals Database. Linda Bourgoyne 1627, R&D Library is available for Global Database searches.
3. Corporate S&LP "Guidelines for Reactive Chemicals Program"
4. Louisiana Division Safety and Loss Prevention Standard S-315 "Reactive Chemicals"
5. Corporate Safety and Loss Prevention Guidelines
6. Reactive Chemicals Self Evaluation Questionnaire (attached)
7. Dow Physical Properties Database
8. NFPA Fire Protection Guide
9. Process Engineering for relief system design assistance.
10. Reactive Chemicals Lab for testing and consultation, Bldg. 2511
11. Members of Louisiana Division Reactive Chemicals Committee.
12. Your department Safety Superintendent.
13. Product Technology Centers
14. Corporate Loss Prevention "Chemical Hazard Engineering Guidelines" 1990 Edition

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SELF EVALUATION QUESTIONNAIRE

1. TESTING AND EVALUATION OF REACTIVE CHEMICAL DATA

- a. Has reactive chemical test data been gathered for the following:

Process materials and mixtures that are normal to the process.
Yes [] No []

Mixtures that may result from abnormal conditions.
Yes [] No []

Material of construction throughout the process. Are wrong materials of construction identified? Yes [] No []

Effects of pH	Yes []	No []
Abnormal temperature	Yes []	No []
Water	Yes []	No []
Abnormal concentrations	Yes []	No []
Flammable dusts	Yes []	No []
Explosive range known	Yes []	No []
Rate of pressure rise data	Yes []	No []

Accelerating Rate Calorimetry (ARC) test on reactive mixtures that are handled in drum or larger volumes.
Yes [] No []

Chemical formulations. Yes [] No []

Changes of chemical components in formulation
Yes [] No []

Significant change in concentrations of components in formulation
Yes [] No []

- b. Have all systems been evaluated with regard to result from test data? Yes [] No []

Has all reactive chemicals data been reviewed by the Reactive Chemicals Test Group and Process Engineering as it relates to the process?
Yes [] No []

Has all data been reviewed by Process Engineering as it relates to pressure relief design?
Yes [] No []

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- c. Do all individuals involved with reactive chemical evaluation understand the information on the data sheets and know the limitations for extrapolating operating parameters from the data?
Yes [] No []
- d. Is process data current? Yes [] No []
- Does it thoroughly define hazardous bounds?
Yes [] No []
- Side reactions? Yes [] No []
- Does data include total potential energy available?
Yes [] No []
- Has test data that was generated before 1/1/82 been reviewed with the Reactive Chemical Testing Lab?
Yes [] No []
- Has irrelevant RC data been discarded? Yes [] No []
- Has reactor pressure relief analysis and design documentation been completed for vessels containing reactive materials?
Yes [] No []

2. CONFIRMING ANALYSIS OF RAW MATERIALS

- a. Is there a program in place for confirming positive identification of all raw materials? (Includes outside vendor and Dow supplied raw materials and also raw materials being supplied by pipeline).
Yes [] No []
- Is a Certificate of Analysis required and received for those difficult to test? Yes [] No []
- b. Are inhibitor levels tested on in-coming raw materials that require an inhibitor? Yes [] No []

3. CRITICAL INSTRUMENTS AND DEVICES

- a. Are critical instruments and devices provided with redundancy?
Yes [] No []
- b. Do you have a program for testing all critical instruments?
Yes [] No []

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- c. Are there any critical instruments in the process that, if failed, would not alarm or indicate that the signal is incorrect? Yes ☐ No ☐
- d. Are alarms activated when critical instruments fail? Yes ☐ No ☐
- e. Is the emergency reactive chemicals functionality of critical instruments documented? Yes ☐ No ☐
- f. Has the process been reviewed within the past two years to determine if adequate monitoring of important parameters is in place? Yes ☐ No ☐
- g. Is there a policy covering changes in set points for trips and alarms? Yes ☐ No ☐

4. MATERIALS OF CONSTRUCTION

- a. Are unwanted reactions that can be caused by material of construction known by those who can install the wrong material? Yes ☐ No ☐
- b. Are there procedures in place to assure that materials such as lubricants, thread compound, valve packing, leak repair compounds, etc. are compatible with the process chemicals? Yes ☐ No ☐
- c. Are completely halogen-saturated lubricants used in Chlorine service? Yes ☐ No ☐
- d. Does the Tech Center provide information/recommendations for material of construction? Yes ☐ No ☐
- e. Is information on material of construction sought from suppliers of raw materials? Yes ☐ No ☐

5. SIDE REACTIONS

- a. Have side and insitu reactions in your process been adequately researched and identified? Yes ☐ No ☐
- b. Have conditions for unwanted reactions been identified? Yes ☐ No ☐

PH Yes ☐ No ☐

Water Yes ☐ No ☐

Contaminants Yes ☐ No ☐

H2 evolution Yes ☐ No ☐

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- c. Is any part of your process subject to peroxide, acetylide or azide formation? Yes [] No []

If so, are the sources known and a program in place to keep this under control? Yes [] No []

6. OPERATING DISCIPLINE

- a. Is Operating Discipline documentation current and complete? Yes [] No []

Has it been reviewed by all operating personnel within the past 12 months? Yes [] No []

- b. Are the Start up and Shutdown sequences written and available for operating personnel? Yes [] No []

Are reasons for sequences and consequences of deviation well documented and understood by operating personnel? Yes [] No []

- c. Is the process designed to avoid the activation of large amounts of reactants together at the same time? Yes [] No []

Is the most critical reactant metered in under carefully monitored rates that are controlled by temperature/pressure/flow parameters? Yes [] No []

Is it possible for a critical operating parameter such as flow to be wrong without the operator knowing? Yes [] No []

Are analytical devices in place to verify critical parameters? Yes [] No []

- d. Are procedures in place to prevent charging of the wrong materials from look-alike drum? Yes [] No []

Are look-alike drums stored in different storage areas? Yes [] No []

- e. Are conditions that approach out-of-range parameters alarmed? Yes [] No []

Is redundant instrumentation and procedures in place to prevent over-filling vessels? Yes [] No []

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SCENARIOS

- a. Are worse case credible scenarios and corrective procedures documented for each step of the process?
Yes [] No []

- b. Have worst case scenarios been highlighted and the process system analyzed for preventive or fail-safe provisions?
Yes [] No []

Do these provide automated protection if the operating personnel fail to take proper action? Yes [] No []

Are the consequences known by operating personnel when equipment and operating errors occur in the same scenario?
Yes [] No []

- c. Do all parts of the process go to a fail-safe mode in a:

Power failure?	Yes []	No []
Cooling loss?	Yes []	No []
Nitrogen failure?	Yes []	No []
Air failure?	Yes []	No []

SCRUBBERS

- a. Is back-flow of the scrubber chemicals into the process possible?
Yes [] No []
- b. Can flammable or reactive materials accumulate in the scrubber medium or vapor space?
Yes [] No []
- c. If the scrubber is pH controlled, are pH monitoring and adjustment features provided?
Yes [] No []
- d. Are additional scrubber gases such as nitrogen purges etc. controlled adequately to prevent loss of scrubber efficiency?
Yes [] No []

9. WASTE WATER STRIPPER

- a. Is the waste water storage vessel protected from the wrong material entering it?
Yes [] No []
- b. Is the stripper protected from back flow or back pressure?
Yes [] No []
- c. Is level monitoring with low level alarm provided for the stripper and any associated knock-out vessels?
Yes [] No []

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10. CATALYSTS

- a. Can catalyst in your process transport downstream and become a hazard? Yes ☐ No ☐
- b. Can iron chloride be made in your process by inadvertent pH excursions? Iron chloride is an effective catalyst for many reactions and has been responsible for many incidents. Yes ☐ No ☐
- c. Are procedures in place for starting up a catalyst or adsorbent bed that prevent a high temperature plug-flow temperature front? Yes ☐ No ☐
- d. Are all potential reactions known for improper flows or conditions across the catalyst bed? Yes ☐ No ☐
- e. Can the catalyst be affected by material of construction, or some ingredient in the formulation? Yes ☐ No ☐
- f. Can an unintended material backflow or migrate into the process and have a catalytic effect? Yes ☐ No ☐

11. INHIBITORS

- a. Are inhibitor levels monitored? Yes ☐ No ☐
- b. Are inhibitors temperature sensitive with respect to effectiveness? Yes ☐ No ☐
- c. Is freeze protection necessary? Yes ☐ No ☐
- d. Is separation of inhibitor from product temperature dependent? Yes ☐ No ☐
- e. Does the inhibitor require a small amount of oxygen to be effective? Yes ☐ No ☐
- If so, are oxygen levels monitored? Yes ☐ No ☐
- f. Is the inhibitor affected by material of construction or vice versa? Yes ☐ No ☐

12. LAB CHEMICAL STORAGE

- a. Is there a policy governing ownership responsibility for lab chemicals? Yes ☐ No ☐

- b. Has the material of construction for the chemical containers been tested for long term compatibility? Yes [] No []
 - c. Is there a receiving procedure to prevent chemicals from being left at a delivery point for excessive periods of time? Yes [] No []
 - d. Is the storage life/self-reactivity/aging data known for stored materials so as to establish a maximum storage age at a given temperature? Yes [] No []
 - e. Are unstable chemicals given special storage provisions? Yes [] No []
- Are stored items in drums checked for over-pressure? Yes [] No []
- Are containers occasionally found to be over-pressured? Yes [] No []
- Is moisture a problem with some chemicals you store? Yes [] No []
- f. Are drums of materials that are sensitive to water stored indoors? Yes [] No []

13. HAZARDOUS MATERIAL HANDLING

- a. Have inadvertent operations been identified which could subject process materials (specifically gases) to heat of compression? Yes [] No []
 - b. Do you vacuum break with nitrogen? Yes [] No []
 - c. Do you monitor for oxygen content in the part of the process that operates under vacuum? Yes [] No []
- Is oxygen monitored in process systems where the components can be easily oxidized? Yes [] No []
- d. Is the design of your process system such that it is virtually impossible for two highly reactive chemicals to get together unintentionally? Yes [] No []
- Are there redundant safeguards? Yes [] No []

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- e. Are drums of materials that are incompatible kept segregated? Yes ☐ No ☐
- Are they identified by label/color code etc.? Yes ☐ No ☐
- f. Are procedures, equipment, and training in place to quickly handle leaks of hazardous materials to prevent exposure to the plant or community? Yes ☐ No ☐
- g. In the event of a leak or spill do you know which absorbent material to avoid? Spontaneous combustion incidents are common. Yes ☐ No ☐
- h. Can material be concentrated or built up in a recycle stream to the point of instability? Yes ☐ No ☐
- Can polymer or other materials in the process build up and become pyrophoric when exposed to air? Yes ☐ No ☐
- i. Are any peroxide-forming materials and conditions present in your process? Yes ☐ No ☐

4. WASTE HANDLING

- a. Is sawdust or cellulosics based materials used as an absorbents? Yes ☐ No ☐
- If so, are procedures and ratios specified to prevent reaction or spontaneous combustion? Yes ☐ No ☐
- b. Have all absorbents in use been tested with the process materials? Yes ☐ No ☐
- Are there any prohibited absorbents? Yes ☐ No ☐
- Do employees know? Yes ☐ No ☐
- c. Are different waste chemicals collected in a common container? Yes ☐ No ☐
- If so, is control of what is put into the container vested in one person or job? Yes ☐ No ☐
- Are pH or other analytical procedures used as part of this control? Yes ☐ No ☐

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- d. Are joint procedures with Environmental Services periodically reviewed? Yes [] No []
- e. Does control of waste include? Yes [] No []
- Material of construction Yes [] No []
- Time limit of storage Yes [] No []
- Written procedure for disposal Yes [] No []
- Ownership Yes [] No []

15. HEELS IN TANK CARS, TANK TRAILERS, BARGES, PROCESS LINES AND STORAGE VESSELS

- a. Are procedures in place to analytically test all heels to assure that the look-alike heels are what they are supposed to be? Yes [] No []
- b. Is odor used as an identifying method? Yes [] No []
- c. Are low points in pipelines checked? Yes [] No []
- d. Are heels from cleaning, such as water, a hazard with your products? Yes [] No []
- e. Are vapors in pressurized vessels/trailers analyzed if no liquid sample can be obtained? Yes [] No []
- f. Are dedicated transport vessels used without confirming analysis of the heels? Yes [] No []

16. EXCHANGERS

- a. Is the exchanger media compatible with the process material? Yes [] No []
- b. Will a leak into the process be hazardous? Yes [] No []
- c. Is monitoring provided to detect a leaking exchanger? Yes [] No []
- d. Is the exchanger essential to the safety of the process? Yes [] No []
- e. Is temperature monitoring provided around the exchanger? Yes [] No []

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- f. Has the maximum temperature of the reboiler been evaluated with respect to length of time exposure, such as during total reflux conditions? Yes [] No []
- g. Have the effects of a leaking steam valve to a shutdown reboiler been evaluated? Yes [] No []
- h. Has the heat history of polymerizable bottoms been evaluated with respect to reboiler temperature and length of time of exposure? Yes [] No []
- i. Have you evaluated the effects of recirculation or cooling failure? Yes [] No []

17. AGITATORS

- a. Is monitoring provided to detect agitator failure? Yes [] No []
 - Motor amps? Yes [] No []
 - Rotation detectors? Yes [] No []
 - Blade detector? Yes [] No []
- b. Has agitator failure been evaluated during the step with the most available energy to determine if a run-away can occur? Yes [] No []
- c. Are reactive feed flows interlocked to stop on agitator failure? Yes [] No []
- d. Are run step procedures in place to assure proper mixing and proper reaction? Yes [] No []
- e. Are amp readings alone on the agitator appropriate to determine the progress of the reaction? Yes [] No []

18. STATIC MIXERS

- a. Are temperatures before and after monitored? Yes [] No []
- b. Are there sufficient reactants within the mixer that overheat could result if flow of reactants is suddenly stopped without purge-out? Yes [] No []

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19. PUMPS

- a. Are feed pumps interlocked with shutdown trips?
Yes [] No []
- b. Are pumps temperature monitored on or within 6-inches downstream of the pump casing to detect deadheading?
Yes [] No []
- c. Does the monitoring scheme assure detection and alarm on pump cavitation?
Yes [] No []
- d. Are pump seals monitored for flow of seal fluid and seal loss?
Yes [] No []
- e. Have seal fluid leaks into the process streams been evaluated?
Yes [] No []
- f. Is temperature monitoring provided for pumps that are used for mixing?
Yes [] No []

20. BACK FLOW

- a. Have potential back flow scenarios been evaluated?
- b. Has process back-flow been effectively prevented in the following?

Feed lines	Yes []	No []
Nitrogen connections	Yes []	No []
Condensate	Yes []	No []
Gas cylinders	Yes []	No []
- c. Are means other than block valves and check valves provided to prevent back flow, such as stopping pumps and depressuring?
Yes [] No []

21. LOW FLOW RATE

- a. Have low flow rates been evaluated for their effects on reaction stability and heat removal? Yes [] No []

Example: throttle back during startup/shutdown/hurricane shutdown.

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- b. Has total reflux of your distillation column or reactor systems been evaluated for loss of overheads through leaking overhead valves? Yes ☐ No ☐

Concentration of unstable components in certain trays
Yes ☐ No ☐

Overheating/heat history problems in the reboiler?
Yes ☐ No ☐

22. RELIEF DEVICES and VENT SYSTEMS

- a. Have pressure relief systems been designed for worse case credible reactive chemicals run-away? Yes ☐ No ☐

- b. Are relief valve discharge headers combined with other vents?
Yes ☐ No ☐

If so, is there a reactive problem? Yes ☐ No ☐

- c. Have hazard analysis been performed and documented to define credible reactive pressure relief scenarios?
Yes ☐ No ☐

- d. Does documentation of reactive pressure relief designs meet minimum requirements as it applies to the following?
Yes ☐ No ☐

Reactive chemicals tests or kinetic data used as a basis
Yes ☐ No ☐

Scenarios used for design. Required capacity
Yes ☐ No ☐

Device size, PSV orifice, manufacturer, serial no., spec sheet
Yes ☐ No ☐

23. COMPATIBILITY CHART

- a. Has a current cross compatibility chart been developed for displaying chemical compatibility of the materials used in the block that may be mixed unintentionally?
Yes ☐ No ☐

- b. If so, are the materials that are self-reactive included in the data? Yes ☐ No ☐

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- c. Is the compatibility chart posted where all block employees can use it? Yes [] No []
- d. Is there a policy for keeping the cross-compatibility chart up-to-date? Yes [] No []

24. REACTIVE CHEMICAL TRAINING

- a. Is periodic reactive chemical training conducted and documented? Yes [] No []
- b. Does reactive chemical training include materials specific to the block? Yes [] No []
- c. Has hazard information of chemical mixtures that can be reactive/flammable/explosive been included in the training program? Yes [] No []
- d. Is the cross-compatibility chart included in the periodic reactive chemical training? Yes [] No []
- e. Are case histories of past incidents in your unit, the Dow world, and the chemical industry that are related to the process you operate included in the training program?
Yes [] No []
- f. Have potentially wrong mixtures or procedures been emphasized in the training program so that employees will know not to allow certain chemicals to mix or to get out of parameters?
Yes [] No []
- g. Are employees trained on the ways instruments and control devices are prone to fail? Yes [] No []
- h. Are employees trained on materials of construction that can cause a potential reactive chemical problem?
Yes [] No []

25. REACTIVE CHEMICAL REVIEWS

- a. Are all processes in the block reviewed by line supervision and engineers, technology center, and Safety Superintendent at least every two years? Yes [] No []
- b. Is a Reactive Chemical Committee Review held for your processes every two years or within 90 days of a new superintendent?
Yes [] No []

- c. Are process changes reviewed by members of the
Reactive Chemicals Committee before they are implemented?
Yes [] No []
- d. Are pre-startup reviews held for process changes and
recommissioning out-of-service equipment? Yes [] No []
- e. Are product formulation changes reviewed by the Reactive
Chemicals Committee? Yes [] No []

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