

TO: Wayne Hilgers

Muriatic Acid

FROM: T. G. Grumbles

DATE: June 9, 1986

Interoffice
Communication

SUBJ: "FOOD GRADE" ACID

VISTA

As we discussed, the Food & Drug Administration (FDA) has yet to finalize it's determination on the proposal to revoke GRAS status for by-product hydrochloric acid. That decision is not on the agency's priority list according to the current project manager at FDA. The proposed specifications for hydrochloric acid, including benzene levels, have been published by the Food Chemicals Codex in a second supplement to the third edition (attached).

Based on the above, and the current manufacturing conditions at Baltimore, I recommend the following statement or portions of the statement be added to the Muriatic Acid technical data sheet or other marketing material.

FOOD AND FOOD PROCESSING APPLICATIONS

Vista has available Muriatic Acid that meets or exceeds the specifications for hydrochloric acid set forth in the Food Chemicals Codex, Edition III, Second Supplement. Specifications can be provided to assist in establishing the suitability of this acid for use in food or food processing applications.

Please let me know if you would like me to prepare a letter or presentation for the "acid marketers" to assure their understanding of the issues involved in the food grade certification question.



Thomas G. Grumbles

ajo/9

Attachment

cc RAK, MJF, PCG, RRC, PTH, THH, OCK, BRBM, DTP

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**SECOND SUPPLEMENT
TO THE
THIRD EDITION**

FOOD CHEMICALS CODEX

COMMITTEE ON FOOD CHEMICALS CODEX

**Food and Nutrition Board
Commission on Life Sciences
National Research Council**

**NATIONAL ACADEMY PRESS
Washington, D.C. 1986**

VVV 000017018

NATIONAL ACADEMY PRESS 2101 CONSTITUTION AVENUE, NW WASHINGTON, DC 20418 ²⁻²

NOTICE The project that is the subject of this report was approved by the Governing Board of the National Research Council, whose members are drawn from the Councils of the National Academy of Sciences, the National Academy of Engineering, and the Institute of Medicine. The members of the Committee responsible for the report were chosen for their special competences and with regard for appropriate balance.

NATIONAL RESEARCH COUNCIL The National Research Council was established by the National Academy of Sciences in 1916 to associate the broad community of science and technology with the Academy's purposes of furthering knowledge and of advising the federal government. The Council operates in accordance with general policies determined by the Academy under the authority of its congressional charter of 1863, which establishes the Academy as a private, nonprofit, self-governing membership corporation. The Council has become the principal operating agency of both the National Academy of Sciences and the National Academy of Engineering in the conduct of their services to the government, the public, and the scientific and engineering communities. It is administered jointly by both Academies and the Institute of Medicine. The National Academy of Engineering and the Institute of Medicine were established in 1964 and 1970, respectively, under the charter of the National Academy of Sciences.

FOOD AND NUTRITION BOARD The Food and Nutrition Board was established in 1940. It is a division of the Commission on Life Sciences of the National Research Council.

The Board serves as an advisory body in the field of food and nutrition. It promotes needed research and helps interpret nutritional science in the interests of public welfare. The Board acts in response to requests from public agencies and, at times, on its own initiative.

The Board is active in areas of dietary guidelines, nutrition and health, food safety, food chemicals specifications, food resources, and international nutrition programs. It has established, among other important guides, recommended dietary allowances, principles and procedures for the evaluation of the safety of foods, specifications of identity and purity for food chemicals, guidelines for nutrient fortification of foods, and recommendations for maternal and infant nutrition. The Food and Nutrition Board draws upon the knowledge and expertise available from the combined resources of academia, government, and industry.

Financial support for the work of the Board is primarily provided by government contracts and grants. In addition, uncommitted support is provided by private foundations and industrial organizations.

Through members of its liaison panels, technical input in aspects of nutrition, food safety, food technology, and food processing is provided.

This study is supported by U.S. Food and Drug Administration Contract No. 223-78-2053 (formerly Grant No. FD 00213).

COMPLIANCE WITH FEDERAL STATUTES The fact that an article appears in the *Food Chemicals Codex* or its supplements does not exempt it from compliance with requirements of acts of Congress, with regulations and rulings issued by agencies of the United States Government under authority of these acts, or with requirements and regulations of governments in other countries that have adopted the *Food Chemicals Codex*. Revisions of the federal requirements that affect the Codex specifications will be included in Codex supplements as promptly as practicable.

LIBRARY OF CONGRESS CATALOG CARD NUMBER 81-38403
INTERNATIONAL STANDARD BOOK NUMBER 0-309-03090-0

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Printed in the United States of America

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fraction, take care to include all saccharides in the standard composition calculation.

Compute the dry-basis concentration (C), in percent, of each individual component in the standard solution by the formula:

$$C = (W_i / \Sigma W_i) \times 100.$$

in which W_i is the weight of the sugar of interest and ΣW_i is the sum of all sugar components. Standardize by injecting 10-20 μ l (about 1.0-2.0 mg solids) of the standard sugar solution. Integrate the peaks and normalize. Sum the individual DP_{4+} responses from the normalized printout to obtain the total DP_{4+} normalized response. Calculate the response factors as follows (see page 476, and page 73 of THIS SUPPLEMENT):

$$R_i = \frac{\text{known concentration, dry basis \%}}{\text{measured concentration, normalized \%}}$$

in which R_i is the response factor for component i .

Compute the response factor for each component relative to glucose (R') using the following equation:

$$R' = R_i / R_G.$$

in which R_G is the response factor for glucose. The R' for DP_{4+} should be programmed as a default value (if automated equipment is used) and used to compute the concentration of higher saccharides.

Sample Analysis Determine the solids content (see below) of the sample and dilute to approximately 10% solids with water. Inject a volume (10-50 μ l) appropriate for the specific solids content.

Calculation Calculate the concentration of each component as follows:

$$C_i = (A_i \times R_i \times 100) / (\Sigma A_i R_i),$$

in which A_i is the area recorded for that component and $\Sigma A_i R_i$ is the sum of the product of the areas (A) and response factors (R) for all components detected.

Arsenic A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Arsenic Test*, using 1 ml of *Standard Arsenic Solution* (1 μ g of As).

Color

Apparatus Use a suitable variable-wavelength spectrophotometer capable of measuring percent transmittance throughout the visible spectrum and designed to permit the use of sample and reference cells with pathlengths of 2-4 cm. The transmittance of all paired cells should agree within 0.5%.

Standard Solution Dissolve 0.10 g of reagent grade potassium dichromate ($K_2Cr_2O_7$) in 1 L of water and mix thoroughly.

Procedure With water in sample and reference cells of 2-cm pathlength adjust the percent transmittance scale of the spectrophotometer to 100%. Leave the reference cell in place and replace the water in the sample cell with the *Standard Solution*; determine the wavelength at which it exhibits exactly 54.5 percent transmittance. This wavelength is defined as λ_1 , the corrected 450-nm wavelength. Remove the 2-cm cells from the spectrophotometer and with water in the sample and reference cells of 4-cm pathlength adjust the

percent transmittance scale to 100% with the spectrophotometer set at λ_1 . Leave the reference cell in place and replace the water in the sample cell with the sample of high-fructose corn syrup. Measure the percent transmittance (T_{450}). Remove the sample cell, set the wavelength at 600 nm, replace the sample with water, and adjust the percent transmittance scale to 100%; then determine the percent transmittance at 600 nm (T_{600}) with the same sample of high-fructose corn syrup in the sample cell. Calculate the *Color* (C) of the sample with the following formula:

$$C = (\log T_{600} - \log T_{450}) / 4,$$

in which T_{600} is the percent transmittance at 600 nm and T_{450} is the percent transmittance at 450 nm.

Heavy Metals Prepare and test a 2-g sample as directed in *Method II* under the *Heavy Metals Test*, page 512, using 20 μ g of lead ion (Pb) in the control (*Solution A*) and 500° as the ignition temperature.

Lead Transfer 10 g of the sample to an evaporating dish, add 5 ml of sulfuric acid solution (1 in 4), mixing it thoroughly with the sample, and evaporate most of the water on a steam bath. Char and dehydrate the sample by heating on a hot plate, while heating at the same time with an infrared lamp from above, and then heat in a muffle furnace at 500° until the residue is free from carbon. Remove the dish from the furnace, cool, and cautiously wash down the inside of the dish with water. Add 1 ml of 1 *N* hydrochloric acid, evaporate to dryness on a steam bath, then add 2 ml of 1 *N* hydrochloric acid, and heat briefly, while stirring, on a steam bath. Quantitatively transfer the solution into a separator with the aid of small quantities of water, and neutralize with 1 *N* ammonium hydroxide. This *Sample Solution* meets the requirements of the *Lead Limit Test*, using 10 g of lead ion (Pb) in the control.

Residue on Ignition Ignite 5 g as directed under *Residue on Ignition*, page 533.

Solids Determine the percent solids from the refractive index (see page 533) as directed under *High-Fructose Corn Syrup Solids*, page 84 of THIS SUPPLEMENT.

Sulfur Dioxide Proceed as directed under *Sulfur Dioxide*, page 546, using a 100 g sample.

Packaging and Storage Store in tight containers.

Functional Use in Foods Nutritive sweetener.

Hydrochloric Acid, page 144

Replace the *Description* and *Requirements* with the following:

DESCRIPTION

A water solution of hydrogen chloride of varied concentrations. It is a clear, colorless or slightly yellowish, corrosive liquid having a pungent odor. It is miscible with water and with alcohol. Concentrations of hydrochloric acid commercially

available are usually expressed in Baumé degrees (Be°) from which percentages of hydrochloric acid and specific gravities can readily be derived (see *Hydrochloric Acid Table*, page 514). The usually available concentrations are 18°, 20°, 22°, and 23° Be. Concentrations above 13° Be (19.6%) fume in moist air, lose hydrogen chloride, and create a corrosive atmosphere. Because of these characteristics, suitable precautions must be observed during sampling and analysis to prevent losses.

Note: Hydrochloric acid is produced by various methods that might impart trace amounts of organic compounds as impurities. The manufacturer, vendor, or user is responsible for identifying the specific organic compounds which are present and for meeting the *Requirements for Extractable Organic Compounds*. Methods are provided for their determination. In applying the procedures any necessary standards should be used to quantitate the organic compounds present in each specific product.

The variety of organic impurities that might conceivably be found in hydrochloric acid is such that it is impossible to provide a comprehensive and accurate list here. Therefore, the manufacturer, vendor, or user is responsible for establishing the suitability of such hydrochloric acid for its intended application in foods or food processing in accordance with the provision on *Trace Impurities*, page 3.

REQUIREMENTS

Identification

... gives positive tests for *Chloride*, page 516.

Assay Within the range of Baumé degrees specified or implied by the vendor.

Arsenic (as As) Not more than 1 mg/kg.

Color Passes test.

Concentration of HCl Within the range specified or implied by the vendor.

Extractable Organic Compounds

Total Organic Compounds (Non-Fluorine-Containing)

Not more than 5 mg/kg, including:

Benzene Not more than 0.05 mg/kg.

Vinyl Chloride Not more than 0.05 mg/kg.

Fluorinated Organic Compounds (total) Not more than 25 mg/kg.

Heavy Metals (as Pb) Not more than 5 mg/kg.

Iron Not more than 5 mg/kg.

Nonvolatile Residue Not more than 0.5%

Oxidizing Substances (as Cl₂) Not more than 0.003%.

Reducing Substances (as SO₂) Not more than 0.007%.

Specific Gravity Within the range specified or implied by the vendor.

Sulfate Not more than 0.5%.

TESTS

Extractable Organic Compounds Proceed as directed under *Extractable Organic Compounds (in Hydrochloric Acid)*, page 80 of THIS SUPPLEMENT.

Insert the following new monograph to precede the monograph entitled *Iron, Carbonyl*, page 151:

Invert Sugar

Invert Sugar Syrup, Invert

DESCRIPTION

Invert sugar is a mixture of glucose and fructose that results from the hydrolysis of sucrose in accordance with good manufacturing practices. Invert sugar is marketed as a component of invert sugar syrup that also contains sucrose in various amounts as represented by the manufacturer.

Invert sugar syrup is a hygroscopic liquid that has a sweet taste, is very soluble in water, glycerin and glycols, and is very sparingly soluble in acetone and ethanol.

REQUIREMENTS

Identification

Prepare a 10% solution in purified water and inject 7.5 µl into a high-performance liquid chromatographic system equipped with a cation exchange resin maintained at 85° and a differential refractometer detector; the liquid phase is purified water eluting at a flow rate of 0.7 ml per min. The chromatogram of the sample gives appropriate elution times for fructose, glucose, and sucrose when compared to a standard solution containing 1 g of each saccharide in 100 ml of purified water.

Assay Sucrose and invert sugar content shall be as represented by the manufacturer.

Arsenic (as As) Not more than 3 mg/kg.

Heavy Metals (as Pb) Not more than 7 mg/kg.

Lead Not more than 5 mg/kg.

pH Not less than 3 nor more than 5.5.

Residue on Ignition Not more than 0.2%.

Total Solids As represented by the vendor.

Total Sugars Not less than 99.5% of the total solids content.

TESTS

Assay

Apparatus Mount a ring support on a ringstand 1-2 in. above a gas burner, and mount a second ring 6-7 in. above the first. Place a 6-in. open-wire gauze on the lower ring to support a 250-ml Erlenmeyer flask, and place a 4-in. watch glass with a center hole on the upper ring to deflect heat. Attach a 50-ml buret to the ringstand so that the tip just passes through the watch glass centered above the flask. Alternatively, a buret with an offset tip may be used in place of a buret with a straight tip extending through the hole in the center of the watch glass. Place an indirectly lighted white surface behind the assembly for observing the end point.

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80 / FCC III-SECOND SUPPLEMENT / General Tests and Apparatus

must be between 0.95 and 1.00 for any single experiment or from accumulated data.

Recalibrate the system after every ten determinations or two days; whichever occurs first.

Sample Preparation Prepare as directed in the individual monograph.

Procedure Inject the volume of *Sample Preparation* as designated in the monograph onto the column. Determine the concentration of intermediates and side reaction products from the peak areas using the slope (m) and intercept (b) calculated under *Calibration* by the equation:

$$A_i = mC_i + b_i$$

in which C_i is the concentration of the unknown in the *Sample Preparation* and A_i its corresponding peak area.

VOLATILE MATTER

Transfer 1.5-2.5 g of colorant, accurately weighed, to a tared crucible. Heat in a vacuum oven at 135° for 12-15 h. Lower the pressure in the oven to minus 125 mm Hg and continue heating for an additional two h. Cover the crucible, and allow to cool in a desiccator. Reweigh the crucible when cool. The loss of weight is defined as the volatile matter.

WATER-INSOLUBLE MATTER

Transfer about 1 g of colorant, accurately weighed, to a 250-ml beaker and add 200 ml of boiling water; stir to facilitate dissolution of the color.

Tare a Gooch crucible equipped with a glass fiber filter (Reeve Angel, No. 5270, or equivalent). Filter the solution with the aid of suction when it has cooled to ambient temperature. Rinse the beaker three times pouring the rinse through the crucible. Wash the filter with water until the filtrate is colorless.

Dry the crucible and filter in an oven at 135° for at least 3 h, cool them in a desiccator and reweigh to the nearest 0.1 mg. Calculate the percent water-insoluble matter (I) by:

$$I = (W_c/W_s) \times 100,$$

in which W_c is the difference in crucible weight and W_s is the sample weight.

Extractable Organic Compounds (in Hydrochloric Acid)

Extractable Organic Compounds Analyses are to be carried out by gas chromatography employing *Vapor Partitioning* or *Solvent Extraction*, depending upon the characteristics of the compound being determined. It is necessary, however, to use

the *Vapor Partitioning Method* for the determination of benzene and vinyl chloride.

Vapor Partitioning Method This method is suitable for the determination of extractable organic compounds at 0.05-100 mg/kg, but is most appropriate for organic compounds with a vapor pressure greater than 10 mm Hg at 25°. Use a gas chromatograph equipped with a flame ionization detector and a 4-m × 2-mm (id) stainless steel column packed with 15% by weight methyl trifluoropropyl silicone (DCFS 1263, or QF-1, or OV-210, or SP-2401) stationary phase on 80/100 mesh Gas Chrom R or the equivalent. A newly packed column should be conditioned at 120° and 30 ml/min helium flow for at least 2 h (preferably overnight) before it is attached to the detector. For analysis the column is maintained isothermally at 105°; the injection port and detector are maintained at 250°; the carrier gas flow rate is set at 11 ml/min; fuel gas flows should be optimized for the gas chromatograph and detector in use. The experimental conditions may be changed as necessary for optimal resolution and sensitivity. The signal to noise ratio should be at least 10:1.

Preparation of Standard Solutions Prepare a standard solution of the organic compounds to be quantitated in hydrochloric acid (known to be free of interfering impurities) at approximate concentrations of 5 mg/kg, or within ± 50% of the concentrations in the samples to be analyzed.

Place a stirring bar in a 1-L volumetric flask equipped with a ground glass stopper and tare the combination. Fill the flask with reagent-grade hydrochloric acid so that no air space is present when the flask is stoppered and determine the weight of the hydrochloric acid. Calculate the volume (V) in μ l of each organic component to be added from the formula:

$$V = (C \times W)/(D \times 1000),$$

in which C is the desired concentration in mg/kg, W is weight of the hydrochloric acid in g, and D is density of the organic compound in mg/ μ l, and 1000 is a conversion factor with the units g/kg. Add the calculated amount of each component to the hydrochloric acid with a syringe (assure that the syringe tip is under the solution surface), stopper the flask and stir the solution for at least 2 h using a magnetic stirrer.

Calibration Treat the standard in the same way as described for the sample under *Procedure* (below). Determine a blank for each lot of reagent-grade hydrochloric acid and calculate a response factor (R) by dividing the concentration (C) in mg/kg for each component by the peak area (A) for that component (subtract any area obtained from the blank sample):

$$R = C/(A - \text{area of blank}).$$

Gaseous compounds present special problems in the preparation of standards. Therefore, to determine response factors for gaseous compounds use the following method, which will be referred to as the *Method of Multiple Extractions*. Dilute a sample of hydrochloric acid known to contain the gaseous compound of interest with an equal volume of water. Draw 20 ml of this solution into a 50-ml glass syringe; then draw 20 ml of air into the syringe, cap with a rubber septum, and place the syringe on a shaker for 5 min. Withdraw 1 ml of the vapor through the septum and inject it into the chromatograph. Expel the vapor phase from the 50-ml syringe, draw in another 20 ml

of air, repeat the extraction, and inject another 1-ml vapor sample into the chromatograph. Carry out the extraction and GC analysis on the same sample of acid a total of six times. For each impurity plot the area (A_n) determined for extraction n against the difference between A_n and the area determined for extraction $(n + 1)$; that is, plot A_n against $[A_n - A_{n+1}]$. The slope of this line is the extraction efficiency (E) for that impurity into the air.

Inject into the chromatograph 1 ml of a 0.1% (by volume) standard gas sample of each impurity in air and determine the absolute factor (F_a) in g per peak area (A) by the following formula:

$$F_a = (M \times 4.0816 \times 10^{-4})/A,$$

in which M is the molecular weight of the compound.

The concentration (C) in mg/kg of the component in the original sample is calculated by the formula:

$$C = (A \times F_a \times 1.6949 \times 10^5)/E,$$

in which A is the peak area corresponding to the compound (as above), F_a is the absolute factor, and E is extraction efficiency. The response factor is then calculated as:

$$R = C/A.$$

Procedure Dilute a 10-ml sample of hydrochloric acid to be analyzed with an equal volume of water. Draw this solution into a 50-ml glass syringe. Then draw 20 ml of air into the syringe, cap with a rubber septum, and place the syringe on a shaker for 5 min. Draw 1 ml of the vapor through the septum and inject it into the gas chromatograph. Approximate elution times in min for some specific organic compounds are as follows:

Methane and acetylene	1.70
Methyl chloride	2.21
Vinyl chloride	2.29
1,1,1-Trichlorofluoromethane	2.62
Ethyl chloride	2.90
Vinylidene chloride	3.20
Methylene chloride	3.64
Chloroform	4.49
1,1-Dichloroethane	4.53
Carbon tetrachloride	4.86
1,1,1-Trichloroethane	5.90
Benzene	6.00
Trichloroethylene	6.22
Ethylene dichloride	6.61
Propylene dichloride	8.41
Perchloroethylene	9.73

Alternate columns may be required to resolve some combinations of components. Methyl chloride and vinyl chloride are resolved by a 3.7-m $\times$ 3-mm (id) squalane column at 45° and a helium flow of 10 ml/min. Chloroform and 1,1-dichloroethane are resolved by a 4-m $\times$ 3-mm (id) DC 550R column at 110° and a helium flow of 12 ml/min.

Calculation Calculate the concentration (C) in mg/kg of each compound by multiplying its corresponding peak area (A) by the appropriate response factor (R) determined in the *Calibration* protocol.

$$C = R \times A.$$

Precision The relative standard deviation at 5 mg/kg should not exceed 15% for five analyses.

Solvent Extraction Method The solvent extraction technique is suitable for the determination of extractable organic compounds at 0.3-100 mg/kg, but is most appropriate for organic compounds with vapor pressures less than 10 mm Hg at 25°. The conditions for the gas chromatograph are the same as for the *Vapor Partitioning* method, except that the column temperature is 120° and the carrier-gas flow is 21 ml/min.

Preparation of Standards Prepare the *Standard Solution* as described under *Vapor Partitioning*.

Calibration Extract a sample of the *Standard Solution* as directed under *Procedure* (below) and inject it into the gas chromatograph. Determine a blank for each lot of reagent-grade hydrochloric acid and perchloroethylene by extracting the hydrochloric acid in the same way as the standard. Calculate a response factor (R) by dividing the concentration (C) in mg/kg for each component by the peak area (A) for that component (subtract any area obtained from the blank sample):

$$R = C/(A - \text{area of blank}).$$

Procedure Accurately transfer 90 ml of the hydrochloric acid sample and 10 ml of perchloroethylene (free of interfering impurities) into a narrow-mouth 4-oz bottle. Place the bottle in a mechanical shaker for 30 min. Separate the two phases (perchloroethylene on the bottom) and inject 3 μ l of the perchloroethylene extract into the gas chromatograph. Approximate elution times in min for some chlorinated organic compounds are as follows:

Vinylidene chloride	2.94
Methylene chloride	3.27
Chloroform	3.83
Carbon tetrachloride	4.07
1,1,1-Trichloroethane	4.50
Trichloroethylene	4.97
Ethylene dichloride	5.26
Propylene dichloride	6.36
Perchloroethylene	6.95
1,1,1,2-Tetrachloroethane	10.12
1,1,2,2-Tetrachloroethane	13.70
Pentachloroethane	16.19

To determine perchloroethylene and higher-boiling impurities, substitute methylene chloride (free of interfering impurities) for perchloroethylene in the extraction step. For higher-boiling impurities such as monochlorobenzene and the three dichlorobenzenes use a 2.74-m $\times$ 2.1-mm (id) stainless-steel column packed with 10% carbowax 20M/2% KOH on 80/100 mesh chromasorb W (acid washed) at 150° and a nitrogen flow of 35 ml/min.

Calculation Calculate the concentration (C) in mg/kg of each compound by multiplying the corresponding peak area (A) (subtract any area obtained from a blank sample) by the appropriate response factor (R) determined in the *Calibration* protocol:

$$C = R \times (A - \text{area of blank}).$$

Precision The relative standard deviation at 5 mg/kg should not exceed 15% for five analyses.

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RF

RF

Notice Letters

June 4, 1986

VISTA

Mr. B. I. Raffle
Supervising Counsel
Environmental & Engineering Group
Conoco Legal Department
P.O. Box 2197
Houston, TX 77252

Certified Mail
Return Receipt Requested

Mr. H. J. Neeld
Director, Environmental Programs
Environmental Conservation
Conoco Inc.
P.O. Box 2197
Houston, TX 77252

Certified Mail
Return Receipt Requested

RE: U.S. v. Conoco (Case No. 83-2518)

Gentlemen:

Pursuant to the Asset Purchase Agreement dated as of July 20, 1984, among E.I. Du Pont de Nemours and Company, Conoco Inc., and Vista Chemical Company, and the Consent Decree entered in the above action, we hereby provide notice of recently-discovered information which may lead to the filing of an Environmental Claim.

On June 3, 1986, the Vista Lake Charles Vinyl Chloride plant experienced a RVD which resulted in VC emissions. Please contact me if you have any questions regarding this matter.

Sincerely,



Thomas G. Grumbles, C.I.H.
Environmental Quality Manager

ajo/9

cc W. L. McClain
Dick Conrad
Ralph Ferrell

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