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Attachment 8

3M Cottage Grove PFC Air Permitting History

Date	Description
3/10/71	MPCA issues permit covering all air emission "exhaustive processes" at Chemolite. (Permit bears a notation added in 1976 indicating that this is the air permit for all of Chemolite.) The "Process Information" section of the permit refers to five "fluorination cells," which produce "organic fluorides." Six stacks were associated with this process. It also notes that two cells were installed in 1954, two in 1957 and the fifth in 1962. Exhaust volume from the cells is noted at 120 scfm, and a handwritten note indicates installation of a scrubber system added to two cells in 1974. Annual operating rate is listed as 6,000 hours.
5/1/74	MPCA issues Permit for 3M Cottage Grove plant. The permit states: "Permittee's Production Equipment are three (3) process reaction vessels used to fluorinate organic compounds. The process emissions are estimated to be 8 lbs of acid vapors from each process vessel." The permit also describes the control equipments as follows: "The air pollution abatement facility ... is a double scrubber system consisting of a counter flow water spray tower scrubber, followed by a caustic/water counter flow spray tower, Teflon lined vent gases lines, and auxiliary equipment." MPCA also notes that "[s]pecifications and calculations submitted by Permittee indicate that all emissions from this facility will be in compliance with all Minnesota Pollution Control Agency Regulations."
6/10/85	3M submits air permit application for a "new fluorochemical distillation system to be installed at 3M's Chemolite plant." The cover letter notes that emissions from this system are fluorochemical vapors, ammonia and acetone." (Emphasis added.) Specifically, the permit application notes estimated emissions of PFOA of 0.97 lb/hr when operating. MPCA later informed 3M that "[b]ecause the organic emissions from the [fluorochemical distillation system] are below 25 tons per year...no construction or operating permit will be required" for the new distillation unit that would emit PFOA.

Date	Description
1/2/87	3M submits an air emissions permit application for the pilot center facility to be built at Cottage Grove. "This application covers four new pilot scale reactor systems and two pilot scale fluorochemicals cells with a caustic scrubber." Application includes a diagram of the fluorochemical cell process (pg. 8). The application includes flow diagrams showing the process equipment. Emissions from the fluorochemical cells are controlled by a caustic scrubber, and emissions from the reactors are reduced by vacuum jest and a vent condenser. The sample emissions calculation indicates that the emissions from the cell based on a "worst case" emission scenario would be less than 3.29 tons per year of inerts and "fluorocompounds in the form of CnFn+2, where n = 1 to 4.
8/14/87	3M submits air emissions permit application for the Specialty Chemicals Division facility at Cottage Grove. Included in the "Description of Facility and Principal Business Activity" section (pg. 3) is a statement that "[t]he principal products produced are adhesives and fluorochemicals." "A typical reactor system has chemicals charged into the reactor. It is then heated and put under vacuum. The vapors are separated using vacuum jest and condenser and are collected in the decanter or receiver. The receivers are used for blending the finished product. ... The reactors range in size from 70 gallons to 6,000 gallons." This section also notes that "[t]here are 19 fluorochemical cells located in Building 15." "Two water scrubbers have been included to reduce the acid emissions from the fluorochemical cells. This type of installation is currently used on other fluorochemical product equipment.. The scrubber is added as a safety precaution because of the acidic nature of HF and also used for acid emission reduction." Pages 6-7 of this application show a flow diagram of a typical fluorochemical manufacturing process. The emissions calculation estimates worst-case emissions of 175 tons/year of off gases from the cells. As in earlier permits, the substances emitted from the cells are "inerts and "fluorocompounds in the form of CnFn+2, where n = 1 to 4." The application also estimates the efficiency of the scrubbers as 99+%.

Date	Description
10/10/89	MPCA authorizes the installation and operation of a cell off-gas scrubber system (consisting of two scrubbers) for Building 15 at the Specialty Chemicals Division at Cottage Grove. This dual scrubber system replaced an earlier scrubber. The Overview section (under "Facility Description" on page 2) notes that Specialty Chemicals produces a variety of fluorochemicals, and that HF is used as a raw material. "The exhaust gas also includes unregulated fluorocarbons that are by-products of the fluorochemical process." The Facility Description also identifies 20 fluorochemical cells, installed prior to 1968, controlled by a packed tower countercurrent water scrubber with a design efficiency of 98.9%.
11/22/89	MPCA authorizes the installation and operation of two water scrubbers for treatment of hydrogen fluoride (HF) exhaust gases in the event of a sudden release from fluorochemical process equipment at 3M's Specialty Chemicals Division at Cottage Grove.
03/07/90	MPCA amends the Cottage Grove plant's permit to allow operation of an alternative pollution control system "on the fluorochemical cells currently in operation at the Specialty Chemical Division of 3M Chemolite." The permit notes that an existing scrubbing system for hydrogen fluoride (HF) will remain in operation. The new system is intended to recover HF for reuse. The permit as amended sets a numeric emissions limit on HF but does not specify limits for other fluorochemical emissions.
06/27/91	MPCA issues air emission permit #23AC-91-I/0-1 for 3M's installation and operation of three new hydrogen fluoride (HF) scrubbers, "which will serve three existing hydrogen fluoride reactor/receiver systems." The permit also notes that the "scrubbers are being installed voluntarily by 3M." Sections 1.2.1-3 identify the emission units as "fluorocarbon workup reactors." The new scrubbers are packed tower countercurrent water scrubbers with a design efficiency >95%.

Date	Description
07/00/93	MPCA issues permit amendment. "Amendment number 2 to this permit allows the company to replace several tanks...and increase the capacity of some of the electrochemical cells by increasing the amperage that each cell can handle." The permit also notes that, while the actual short term emission rate for HF will increase, the allowable emissions will not change.
04/17/95	3M submits part 70 permit application for the Cottage Grove Specialty Materials Division. Form GI-01, Facility Information, indicates that there are still 19 fluorochemical cells in Building 15 in which fluorinated hydrocarbons are manufactured. Form IA-01 lists fluoride emission rates for specific emissions units. Form GI-02 provides process flow diagrams, including for the emissions units associated with fluorochemical production. These diagrams indicate that the Building 15 fluorochemical cell and reactor systems continue to be controlled by scrubbers. Further, the reactors in Building 70, the pilot facility, are also scrubber-controlled. Form GI-03 provides facility and vent diagrams. Form GI-07 lists fluoride emissions.
06/17/96	3M notifies MPCA of its intention to burn nonhazardous fluorinated gas at the Cottage Grove incinerator.
01/24/97	3M notifies MPCA of additional details of plans to burn fluorinated gases and liquids through the aqueous burner system at the Cottage Grove plant incinerator. Notification indicates that some of the liquids may be hazardous due to the presence of acetone.

Date	Description
07/24/97	MPCA issues a combined permit for the Cottage Grove chemical facility. Page 3 of the permit states that "[t]his permit authorizes the operation of a series of electrochemical cells emitting Hydrogen Fluoride (HF) and a cell off-gas scrubber system." The covered activities were previously permitted under Permit 23AC-96-I/O-I0 issued October 10, 1989 and amended March 7, 1990 and July 21, 1993. The new permit consolidated those documents and increased the emissions limit for SO_2. The permit addresses Stack/Vent 226 (scrubber exhaust), 234 (mist eliminator exhaust), Units 16-19 (reactor systems consisting of electrochemical cells and various tanks), and any emission unit (new or existing) with an Insignificant Modification under Minn. R. 7007.1250. Testing was required with regard to liquid feed material exhausting to the HF recovery system.
6/22/1999	3M submits Request to MPCA to Void Air Emission Permit: Permit No. 23AC-96-I/O-10 - Operation of an Electrochemical Cell System. The reason 3M seeks to void the permit is described as "the electrochemical cells have been permanently shut down. The scrubbers associated with the cells are still in service for other purposes not specified or mandated in this permit."
10/15/1999	3M submits application for the addition of three scrubbers in Building 70 of the Process Development Center at Cottage Grove. The primary purpose of these three scrubbers will be to control fluoride emissions of a pilot sized electrochemical cell system that will begin operation sometime in November, 1999. The emissions for these new electrochemical cells were submitted as insignificant modifications in a letter to MPCA dated April 1997. "Please note that there are no permits or regulations requiring the installation of these scrubbers, but are being installed voluntarily."
12/28/00	3M submits application for the addition of a new baghouse in Building 25 at 3M Cottage Grove Center. The primary purpose of this baghouse is to control the particulate matter (PM) emissions from existing emission units. The letter notes that there are no permits or regulations requiring the installation of this baghouse, but is being installed voluntarily. Attached is MPCA Permit Amendment Application Form - MCP-01 over Page AQD Facility ID No.: 16300022

Date	Description
6/6/2003	3M submits a total facility operating permit re-application. The description states that "[m]anufacturing operations at this site consist of reactor systems ranging in size from 100 gallons to 4000 gallons, and all associated equipment required to batch synthesize a large array of chemicals." "This facility also houses SMMD's Process Development Center (PDC). The PDC is a research and pilot plant operation, with batch reactors ranging in size from 5 gallons to 200 gallons. ... Production for sale into commerce is done at a de minimis level in the PDC."
8/19/2003	3M submits renewal request to use alternate emission calculations for its air emissions inventory. The request application states that 3M's production involves batch chemical operations like reaction, condensation, decanting, distillation, etc. Standard emission factors do not exist for the specific batch chemical reaction processes that occur at this facility. Instead, specific emission factors are calculated for each product manufactured at this facility using a software program entitled EmissionMaster. This program calculates emissions from batch processes using the equations and methods required by EPA in all chemical manufacturing Maximum Achievable Control Technology (MACT) standards promulgated since 1998. This request proposed that 3M continue to use EmissionMaster software to calculate emissions from the operations at 3M Cottage Grove SMMD. This method is used to calculate emissions from all sources at this facility, with the exception of ECF operations in Building 70. These are semi-continuous operations, and not well suited for modeling in a batch-oriented software system. No generally available emission factors exist for these 3M-specific operations. Emissions from ECF processes are determined by completing material balances around the processes at the laboratory level, including analysis of off-gases via IR and/or GC, before moving operations to Cottage Grove.

Date	Description
6/29/2004	This permit authorizes 3M to operate 3M's Specialty Materials Manufacturing Operations located at 3M Cottage Grove Center, which manufactures a large variety of products and fine chemicals. Many of these are used by other 3M divisions as intermediates to produce adhesives and fluorochemicals. This facility is co-located with Chemical Process Development Center (CPDC), which is a research and development. The majority of the emission units at the facility are reactors or reactor systems. These emission units contribute primarily VOC and HAP emissions. Some of the other non-reactor emission units are: grinders, mixers, mills, holding tanks, electric ovens, vacuum ovens, mix tanks, process tanks, columns, centrifuges, boilers, and emergency generators. This facility has pollution control equipment controlling PM, PM10, SO2, VOCs, and HAPs. 3M requested that one control device (CE 019 - gas scrubber) be included in the permit, and hence this permit contains requirements for CE 019, controlling Fluoride emissions.

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Attachment 9

Bibliography of Articles within 3M's Possession Related to Fluorochemical Atmospheric Fate & Transport[1] (as of November 13, 2007)

A. Kaiser, M, et al., *Physicochemical Properties of Fluorotelomer Alcohols*, (Abstract only), undated

B. Mabury S, et al., *Atmospheric Degradation Mechanisms of Fluorotelomer Alcohols as Potential Source of Perfluorinated Acids to Remote Regions*, (Abstract only), undated

C. Matthies, KO, Klasmeier J, and Wegman F, *Evaluation of Environmental Exposure and Long-range Transport Potential for Perfluorinated 8-2 Telomer B Alcohol*, (Abstract only), undated

D. Smithwick, M, et al., *Geographical and Temporal Trends of Perfluorinated Surfactants in Polar Bear Liver and Plasma Samples from the North American and European Arctic*, (Abstract only), undated

E. Tromp, TK, et al., *Potential Accumulation of a CFC-replacement Degradation Product in Seasonal Wetlands*, Nature 376: 327-330, July 27, 1995

F. Boutonnet, JC, et al., *Environmental Risk Assessment of Trifluoroacetic Acid*, Human and Ecological Risk Assessment 5(1):59-124 Vol. 1, 1999

G. Ellis, DA, et al., *Atmospheric Lifetime of Fluorotelomer Alcohols*, Environmental Science & Technology 37(17):3816-3820, 3816-3820, 2003

H. Stock, NL, et al., *Polyfluorinated Telomer Alcohols and Sulfonamides in the North American Troposphere*, Environmental Science Technology 38(4):991-6, Feb. 15, 2004

I. Ellis, David A., et al., *Degradation of Fluorotelomer Alcohols: A Likely Atmospheric Source of Perfluorinated Carboxylic Acids*, Environmental Science & Technology 38(12):3316 - 3321, 2004

J. Hurley, MD, et al., *Atmospheric Chemistry of 4:2 Fluorotelomer Alcohol (CF3(CF2)3CH2CH2OH): Products and Mechanism of Cl Atom Initiated Oxidation*, J. Phys. Chem. A, 108:5635-5642, 2004

K. Lei, YD, et al., *Determination of Vapor Pressures, Octanol-Air, and Water-Air Partition Coefficients for Polyfluorinated Sulfonamide, Sulfonamidoethanols, and Telomer Alcohols*, J. Chem Eng. Data, 49:1013-1022, 2004

[1] 3M does not necessarily concur with the conclusions or hypotheses set forth in at least some of these publications. However, the Company is submitting this information as part of the available scientific information.

L. Stock, NL, et al., *Vapor Pressures of the Fluorinated Telomer Alcohols -- Limitations of Estimations Methods*, Environmental Science & Technology 38(6):1693-1699

M. Deon, JC, et al., *Atmospheric Fate of N-Methyl Perfluorobutane Sulfonamidoethanol (N-MeFBSE, C4F9SO2N(CH3)(CH2CH2OH)*, (Abstract only), Presented at FLOUROS Conference, Aug. 2005

N. Ellis, DA, et al., *The Degradation of Fluorotelomer Alcohols in the Troposphere*, (Abstract only), Presented at FLOUROS Conference, Aug. 2005

O. Goss, KU, et al., *Contributing to a Better Understanding of the Partition Behavior of Fluorinated Alcohols and Olefins*, (Abstract only), Presented at FLOUROS Conference, Aug. 2005

P. Korzeniowski, SH., et al., *Sources of Perfluorocarboxylic Acids in the Environment*, (Abstract only), Presented at FLOUROS Conference, Aug. 2005

Q. Martin, JW, et al., *Atmospheric Lifetime and Oxidation Products of N-Ethyl Perfluorobutylsulfonamide (C4F9SO2N(H)CH2CH3)*, (Abstract only), Presented at FLOUROS Conference, Aug. 2005

R. Matthies, M, and Klasmeier, J, *Evaluation of the Environmental Fate and Exposure for Perfluorinated Telomer B Alcohols*, (Abstract only), Presented at FLOUROS Conference, Aug. 2005

S. van Roon, A, and Voogt, P, *Vapour Pressures of Fluorotelomer Alcohols*, (Abstract only), Presented at FLOUROS Conference, Aug. 2005

T. Young, CJ, et al., *Atmospheric Flux of Perfluorinated Acids into the High Arctic*, (Abstract only), Presented at FLOUROS Conference, Aug. 2005

U. Bossi, Rossana, et al., *Preliminary Screening of Perfluorooctane Sulfonate (PFOS) and Other Fluorochemicals in Fish, Birds And Marine Mammals from Greenland and the Faroe Islands*, Environmental Pollution 136:323-329, 2005

V. Kannan, Kurunthachalam, Se Hun Yun, and Thomas J. Evans, *Chlorinated, Brominated, and Perfluorinated Contaminants in Livers of Polar Bears from Alaska*, Environmental Science & Technology 39(23), 2005

W. Prevedouros, Konstantinos, et al., *Sources, Fate and Transport of Perfluorocarboxylates*, Environmental Science and Technology (Manuscript), 2005

X. Emmett, Edward A., et al., *Community Exposure to Perfluorooctanoate: Relationships Between Serum Concentrations and Exposure Sources*, JOEM, Vol. 48, No. 8, Aug. 2006

Y. Martin, J.W., et al., *Atmospheric Chemistry of Perfluoroalkanesulfonamides: Kinetic and Product Studies of the OH Radical and Cl Atom Initiated Oxidation of N-Ethyl Perfluorobutanesulfonamide*, Environmental Science & Technology 40:864 - 872, 2006

Z. Wallington, T.J., et al., *Formation of $C_7F_{15}COOH$ (PFOA) and Other Perfluorocarboxylic Acids during the Atmospheric Oxidation of 8:2 Fluorotelomer Alcohol*, Environmental Science & Technology 40:924 - 930, 2006

AA. Young, Cora J., et al., *Perfluorinated Acids in Arctic Snow: New Evidence for Atmospheric Formation*, Environmental Science & Technology (E-published version), Mar. 28, 2007

BB. Butt, Craig M., et al., *Rapid Response of Arctic Ringed Seals to Changes in Perfluoroalkyl Production*, Environmental Science & Technology 41(1): 42 - 49, 2007

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Attachment 10

Cottage Grove PFC Manufacturing Plant
Industrial Hygiene Sampling

DATE	MATERIAL SAMPLED	CONCENTRATION (MG/M3)
7/20/1977	PFOA-based	0.03 - 0.07
08/02/1977	PFOA-based	3.4
08/10/1977	PFOA-based	0.05 - 33.9
12/02/1977	PFOA-based	ND - 1.0
12/22/1977	Unspecified	0.21, 1.37, 9.28
07/12/1978	PFOA-based	0.06 - 2.3, <0.01 - 0.52
09/11/1978	PFOA-based	0.01 - 0.07
02/09/1979	PFOA-based	0.04
04/11/1979	PFOA-based	0.04
08/10/1979	PFOA-based	0.07 - 2.1
10/00/1979	PFOA-based	0.04 - 0.95, 0.03 - 0.3
01/15/1980	Unspecified	0.03 - 0.54
05/01/1980	PFOA-based	0.01 - 12.8
05/05/1980	PFOA-based	0.05 - 0.24
06/04/1980	PFOA-based	0.1, 0.02 - 0.13, <0.01 - 0.13
06/06/1980	PFOA-based	0.11 - 0.12
06/30/1980	PFOA-based	<0.01 - 0.05
07/03/1980	PFOA-based	<0.01 - 0.19
07/18/1980	Unspecified	0.1 - 1.0
08/11/1980	PFOA-based	0.01 - 0.15
12/19/1980	PFOA-based	0.03 - 0.17
03/25/1981	PFOS-based	0.02
05/15/1981	PFOA-based	0.06 - 0.10
05/18/1981	PFOA-based	0.08 - 6.70
05/19/1981	PFOA-based	0.07 - 0.18
05/20/1981	PFOA-based	0.37
05/21/1981	PFOA-based	0.03 - 2.28
10/20/1981	PFOS-based	<0.01 - 0.02
09/13/1982	Unspecified	0.003 - 0.053
06/21/1984	Unspecified	0.001 - 0.033
06/15/1984	PFOA-based	<0.004 - 0.055
07/03/1984	PFOA-based	<0.002 - 0.043
07/18/1984	PFOA-based	1.03, <0.002 - 0.008
10/03/1984	PFOA-based	0.02 - 0.36
11/20/1984	PFOA-based PFBA-based	<0.003 - 0.08 <0.02 - 70.2

DATE	MATERIAL SAMPLED	CONCENTRATION (MG/M3)
11/29/1984	PFOS-based	0.06 - 0.19
02/15/1985	PECHS-based	0.22 - 0.73
04/26/1985	PFOS-based	0.003 - 0.007
05/01/1985	Unspecified	0.02
07/24/1985	Unspecified	0.02 - 0.15
08/29/1985	PFOA-based	0.002 - 0.003
04/21/1986	PFOA-based	0.012 - 0.033
04/22/1986	PFOA-based	0.008 - 0.11
05/01/1986	PFOA-based	0.005 - 0.130, 0.0104 - 0.03
05/08/1986	PFOA-based	0.004 - 0.12, 0.26 - 1.54
03/00/1990	PMSF-based	0.05
04/10/1992	PFOA-based	0.08 - 0.1
04/13/1992	PFOA-based	<0.002 - 0.05
09/01/1993	PFOS-based	0.04 - 1.42
10/07/1993	PFOS-based	0.45
08/15/1994	PFOA-based	0.004 - 0.15
11/15/1995	PMSF-based	<0.007 - 0.007
09/06/1995	PFOA-based	<0.002 - 0.65
05/12/1998	PMSF-based	0.13 - 0.34
09/16/1998	PMSF-based	<0.019 - 0.593
07/06/1998	PFOS-based PECHS-based	0.0001 - 0.00159, 16 - 4941 0.00281 - 0.04367
08/21/1998	PFOS-based	0.0001 - 0.2536
02/00/1999	PFOS-based	0.024 - 3.7, <0.0042 - <0.0047, <0.0042 - <0.0047
09/18/1999	PFOA-based	0.0056 - 0.24
07/00/2000	PMSF-based PFOS-based PFOA-based	<0.0002 - <0.0007 <0.0002 - 0.0009 <0.0002 - 0.0032
00/00/2000 (various dates)	PFOA-based	<0.4, <4.0, 0.032 - 0.11
01/09/2002	Unspecified	<0.0003 - 0.012
02/00/2002	Unspecified	<0.0003 - 0.0025

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Attachment 11

Summary of Off-Site Waste Disposal Locations

Cottage Grove Site

Facility ID	Waste Disposed	Est. Period of Use	Priority for Investigation	Comments
Oakdale Site	Liquid and solid industrial waste	1956-1960	High	Investigation underway
Woodbury Site	Liquid and solid industrial waste	1960-1966	High	FC wastes remain buried at the site
Great Northern Oil Co plant, in Bruno, MN	Wastewater/ phenol waste	1965	Low	Waste was not FC-related
Kerrick Site, Pine County, MN*	Liquid and solid industrial waste	1967 & 1969	Low	Low waste volume was disposed in this area and this volume has been removed and incinerated
Commercial Chemical Company in Newport, MN (incinerator)	Wet scrap	1960-1962 (pick up of solvent) 1962-1964 (incineration)	Medium	FC-wastes may have been managed at this facility
Commercial Chemical Company/Pollution Control, Inc. in Savage, MN (incinerator)	Wet scrap	1964-1971	Medium	FC-wastes may have been managed at this facility
Pig's Eye Dump	Dry scrap; burned out drums and inert ash from Chemolite incinerator	1971	Low	Pigs Eye primarily was used for dry scrap disposal and one-time disposal of incinerator residue from July 1971 to December 1971

Facility ID	Waste Disposed	Est. Period of Use	Priority for Investigation	Comments
Washington County Landfill (Lake Jane Landfill)	WWTP sludge, incinerator scrubber sludge, iron oxide sludge, and ash from quench chamber of incinerator	1971-1974	High	Investigation underway
Pine Bend Landfill, IGH, MN - BFI (aka Phoenix) Former NPL Site	WWTP sludge and boiler ash	Mid 1970s/ Early 1990s	Medium	WWTP sludge was managed at this facility.
Anoka County (*Uncertain of other names)*	WWTP sludge	1980s	Medium	WWTP sludge may have been managed at this facility.
Rosemount - SKB (USPCI/Laidlaw) (3-4 mi away from Pine Bend)	WWTP sludge and nonhazardous industrial waste from demolition of old incinerator in 2002	Mid 1990s to mid-2003	Medium	WWTP sludge was managed at this facility.
Superior FCR - ONYX Buffalo, MN	WWTP sludge	Mid-2003 to present	Low	WWTP sludge was disposed at this facility after cessation of FC production.

*Materials recovered and brought back to incinerator for disposal