40 CFR Parts 795 and 799

[OPTS-42073A; FRL 3441-8]

2-Mercaptobenzothiazole; Final Test Rule

AGENCY: Environmental Protection Agency (EPA).

ACTION: Final rule.

SUMMARY: EPA is issuing a final test rule, under section 4 of the Toxic Substances Control Act (TSCA), requiring manufacturers and processors of 2-mercaptobenzothiazole (MBT, CAS No. 149-30-4) to perform testing for persistence and mobility, chronic aquatic toxicity, developmental toxicity, reproductive toxicity, neurotoxicity, and mutagenic effects in the dominant lethal assay.

DATE: In accordance with 40 CFR 23.5, this rule shall be promulgated for purposes of judicial review at 1 p.m. eastern daylight time on September 21, 1988. These regulations shall become effective October 21, 1988.

FOR FURTHER INFORMATION CONTACT: Michael Stahl, Acting Director, TSCA Assistance Office (TS-799), Office of Toxic Substances, Rm. EB-44, 401 M Street SW., Washington, DC 20460, (202) 554-1404. TDD: (202) 554-0551.

SUPPLEMENTARY INFORMATION: EPA is issuing a final test rule under section 4(a) of TSCA to require health effects, chemical fate and environmental effects testing of MBT.

Public reporting burden for this collection of information is estimated to average 535 hours per response, including time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding the burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Chief, Information Policy Branch, PM-223, U.S. Environmental Protection Agency, 401 M Street, Washington, DC 20460; and to the Office of Information

and Regulatory Affairs, Office of Management and Budget, Washington, DC 20503.

I. Introduction

A. Test Rule Development Under TSCA

This final rule is part of the overall implementation of section 4 TSCA (Pub. L. 94-469, 90 Stat. 2003 *et seq.*, 15 U.S.C. 2601 *et seq.*), which contains authority for EPA to require the development of data relevant to assessing the risk to health and the environment posed by exposure to particular chemical substances or mixtures (chemicals).

Under section 4(a)(1) of TSCA, EPA must require testing of a chemical to develop health or environmental data if the Administrator makes certain findings as described in TSCA under section 4(a)(1) (A) or (B).

Detailed discussions of the statutory section 4 findings are provided in the Agency's first and second proposed test rules, which were published in the Federal Register of July 18, 1980 (45 FR 48510) and June 5, 1981 (46 FR 30300).

B. Regulatory History

The Interagency Testing Committee (ITC) designated MBT for priority testing consideration in its 15th Report, published in the Federal Register on November 29, 1984 (49 FR 46931). The ITC recommended that MBT be considered for chemical fate testing, including dissociation constant, persistence in water and soil, and leaching and migration; and environmental effects testing, including acute and chronic toxicity to fish, aquatic invertebrates and plants, and terrestrial plants.

EPA responded to the ITC's recommendations for MBT by issuing a proposed rule, published in the Federal Register of November 6, 1985 (50 FR 46121), which would require that MBT be tested for oral and dermal pharmacokinetics, developmental toxicity, reproductive toxicity, neurotoxicity, mutagenic effects (chromosomal aberrations), aerobic aquatic biodegradation, indirect photolysis screening level test, chemical mobility, fish chronic toxicity, and daphnid chronic toxicity.

The proposed rule contained a chemical profile of MBT, a discussion of EPA's TSCA section 4(a) findings, and the proposed test standards.

II. Response to Public Comments

The Agency received written comments on the MBT proposed rule from the Rubber Manufacturers Association (RMA), Dow Chemical Company, and the Rubber Additives Program Panel (RAPP) of the Chemical Manufacturers Association (CMA). A public meeting was also requested by CMA and held on April 14, 1986. The comments received by the Agency in response to the MBT proposed rule are discussed below.

A. Justification for the Substantial Exposure (Section 4(a)(1)(B)) Finding

1. *Production/use.* RMA provided the Agency with comments and additional information, including results of a survey, regarding exposure to and current use of MBT. The presented survey results indicate that consumption of MBT as a vulcanization accelerator is 650,000 pounds per year and that MBT is not typically used in high volume products such as tires, or in consumer products. Based on this survey, the RMA questioned "* * * whether exposure to MBT is sufficiently widespread and substantial to support issuance of a test rule."

The RAPP survey confirms EPA's findings that MBT is now used as an accelerator primarily in certain specialty products. However, the survey fails to indicate that the major use of MBT is in the manufacture of several other rubber accelerators, e.g., NaMBT, ZnMBT, 2,2-dithio-bisbenzothiazole (MBTS), *N-tert*-butyl-2-benzothiazolesulfenamide (BBTS), *N*-cyclohexyl-2-benzothiazolesulfenamide (CBS), and *N*-oxydiethylene-2-benzothiazolesulfenamide (OBTS) (Ref. 1). The total amount of MBT consumed in production of these derivatives will greatly exceed the amount of MBT the RMA indicates will be consumed in production of specialty rubber products, or the 1983 sales volume of 5,958,000 pounds (Ref. 2). Considering the large volumes of MBT used widely in both chemical manufacture and as a specialty rubber accelerator, the Agency maintains that potential for substantial exposure to MBT may exist in the workplace, in consumer goods, and in receiving environments. Furthermore, the MBT-derived rubber accelerators have a strong tendency to decompose in the vulcanization procedure and can form MBT (Ref. 3), which may carry over into end-products and waste effluents. Also, introduction of the MBT salts into the environment can lead to the presence of MBT by simple hydrolysis. Thus, the Agency maintains that when the total pattern of MBT usage is considered, sufficient potential for substantial exposure to MBT exists to support testing requirements under section 4(a)(1)(B) of TSCA.

2. *Exposure data/monitoring.* RAPP also maintained that its survey of national surface waters (18 sites, predominantly in the middle and eastern United States) shows that MBT is not a widespread aquatic contaminant because no MBT was detected at a stated limit of detection of 10 ppb.

EPA continues to believe that this study was limited by several experimental deficiencies, such that meaningful interpretation of the results is difficult. The principal shortcoming of the study was the wide variation in recovery of MBT from field-spiked samples. Compounding this analytical deficiency is the choice of sampling locations, which generally were very large bodies of water (such as the Great Lakes), far removed from sites that manufacture or process MBT. In addition, although some sample sites were in areas of heavy industry (i.e., Mississippi River at Memphis, TN, and Alton, IL; Missouri River at St. Louis, MO; and eastern Lake Erie near Erie, PA), these sites had zero percent recoveries of MBT in field-spiked samples, suggesting that any MBT present in study samples would also have not been detected by the procedures used. In contrast to the above data, that has been detected in several sites associated with the production/processing of MBT (Refs. 26 through 28).

3. *Exposure data/tire dust.* RAPP also commented that the Agency may have overestimated the release of MBT to the environment via leaching from tire dust created by the wear of tires made with MBT. Among the reasons given were: (1) MBT accounts for less than 10 percent of accelerator used in tires; (2) most MBT is bound in the rubber matrix and leaches slowly, if at all, from tire dust contacting water; (3) current tire wear is much longer than when the original EPA estimate was made; and (4) "* * * MBT is too unstable on the terrestrial soil to remain as MBT for long * * *."

While the Agency agrees that the EPA estimate of 12 million pounds of vulcanization accelerators (including MBT) released annually to the environment (primarily soils near highways) via deposition of tire dust containing accelerators may be an overestimate due to the greater life of current tires, the Agency maintains that sufficient questions remain about the other reasons cited by RAPP that this scenario should remain as a cause for exposure concern.

First, the original EPA estimate does not state that all tires are made with MBT. The major use of MBT is in the manufacture of other rubber accelerators, which have a strong tendency to decompose in the vulcanization process, and can form MBT and carry over into end products and waste effluents, considerably broadening the scope of potential

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exposure (Ref. 1). Second, insufficient data exist to conclude that all MBT is tightly bound in the rubber matrix. The study cited by the RAPP to demonstrate only a limited leaching of MBT from tire rubber may actually suggest that MBT leaching can be significant when consideration is given to the experimental design—a simple static water/tire system. In this system, a rapid equilibrium level of MBT in the water was achieved. If the rubber had been exposed periodically to fresh aqueous media, much more MBT could potentially have been leached from the rubber. Finally, there are no data from which to conclude that MBT will not persist in roadside soils following deposition via tire dust. Furthermore, consideration must be given to the possibility that MBT entering soils by this mechanism may reach drainage systems via runoff and subsequently reach nearby surface waters.

4. *Exposure data/occupational and consumer.* RAPP has indicated that EPA lacks evidence of substantial worker exposure to MBT, citing the estimate of the National Occupational Exposure Survey (NOES; Ref. 4) of 2,398 exposed workers, and the use of protective equipment and good hygiene to limit exposure, particularly because of the allergenic properties and disagreeable odor of MBT.

While the Agency agrees that the warning properties of MBT encourage the limitation of work exposure, EPA does not believe that a disagreeable odor and voluntary use of protective equipment will necessarily limit exposure in all occupational scenarios, nor does EPA consider the NOES estimate of 2,398 exposed workers to constitute an insubstantial number. Also, this number may be an underestimate, because the NOES covered only those products containing the chemical name on the product label, not trade name products where substantial worker exposure could occur.

RAPP also commented that MBT use in Spandex-containing clothing has been discontinued. EPA was aware of this at the time the proposed rule was issued (Ref. 1).

5. *Exposure data/chemical fate.* RAPP commented that chemical fate testing, particularly indirect photolysis, of MBT is unwarranted based on lack of substantial environmental occurrence. EPA finds this conclusion to be unjustified, as discussed in Unit II.A.2. of this preamble, due to the deficiencies of the RAPP monitoring study. Chemical fate and persistence data for MBT is necessary to support an Agency risk assessment of MBT.

B. Justification for Hazard Potential: Environmental Effects

RAPP maintains that by applying a 1,000-fold safety factor to acute toxicity data, sufficient environmental effects data exist to predict that chronic toxicity to sensitive aquatic species to MBT at predicted environmental concentrations will not occur. RAPP notes that its environmental monitoring data and "worst case" modeling indicate that environmental water concentrations will not exceed 1 ppb, nearly three orders of magnitude less than the lowest documented acute effects level for fish (670 ppb). RAPP also noted that a previous EPA proposed rule for phenylenediamines (PDAs) allows use of a 100-fold safety factor over predicted environmental concentration to assess the need for chronic effects testing, provided that acute effects data are available for at least three species (51 FR 472; January 6, 1986).

The Agency considers the cited environmental monitoring data, however, to be inadequate as discussed in Unit II.A.2. of this preamble. The "worst case" predicted concentration cited by RAPP of less than 1 ppb is based on uniform dispersal of released MBT to all United States surface waters. In contrast, the Agency has estimated that MBT surface water concentrations near manufacturing sites could range from 2.96 to 385 ppb (Ref. 5), using confidential production and release data submitted by the manufacturers.

Acute toxicity data are available for three fish species (Refs. 6 through 8), an invertebrate (Refs. 9 and 10), and an alga (Refs. 11 and 12). The trout data were obtained under flow-through conditions, with the MBT concentrations periodically measured. All other studies adhered to standard static screening protocols, with no measurement of test material concentrations.

With regard to the need for chronic toxicity testing, EPA, in the Notice of Proposed Rulemaking (NPR) (51 FR 472; January 6, 1986) for PDA's, proposed that fish early life stage tests or daphnid life-cycle tests (chronic toxicity tests) should be conducted if any LC50 or EC50 was less than or equal to 100 times the predicted environmental concentration (PEC). All of the MBT EC50 or LC50 values are less than 100 times the median or maximum PEC. The algal EC50 value is less than 100 times the minimum PEC. In the 2,6-di-*tert*-butyl phenol NPR (52 FR 23862, June 25, 1987), the Agency proposed an additional decision criteria for determining whether chronic toxicity tests should be conducted, *viz.*, EC50 or LC50 less than 1 mg/L. The MBT 96-hr LC50 values for rainbow trout are both less than 1 mg/L.

In the tributyl phosphate NPR (52 FR 43346, November 12, 1987), the Agency proposed that chronic toxicity tests should also be conducted if the ratio of the 24-hr to 96-hr LC50 (for 4-day acute tests) or the 24-hr to 48-hr LC50 (for 2-day acute tests) was greater than or equal to 2 and if the EC50 or LC50 was less than or equal to 150 mg/L. For MBT, the bluegill 96-hr LC50 is less than 100 mg/L and the 24-hr to 96-hr LC50 ratio is greater than 2. Thus, MBT satisfies the criteria originally proposed as well as those subsequently proposed for other chemical substances for conducting chronic toxicity tests. These decision criteria were proposed in the reproposed PDAs NPR (53 FR 913, January 14, 1988).

C. Health Effects

1. *Need for testing/testing scheme.* RAPP noted that the ITC in its initial review of MBT did not recommend health effects testing. It also suggested that it would be prudent to wait for the evaluation of the results of ongoing (pharmacokinetic) and completed (bioassay) tests prior to initiating further health effects studies, and that any further testing considered necessary should be conducted using a tiered approach. A tiered scheme was proposed by RAPP.

In response to these comments, EPA notes that the report prepared by the ITC was a preliminary assessment of the potential need for further testing of MBT. EPA's proposed requirements for health effects testing of MBT were a consequence of a tiered approach to evaluate the need for testing of existing chemicals. This does not imply any inconsistency with the ITC recommendation but rather the evolution of a testing strategy as more information has become available. The Agency has considered the NTP chronic study in the development of the test rule for MBT, and is not requiring further chronic and oncogenicity testing. The design of the particular chronic bioassay performed by NTP, however, is not adequate to answer all toxicological questions of concern.

2. *Developmental toxicity testing.* RAPP maintains that there is insufficient evidence to justify the proposed requirement for testing MBT for teratogenic potential in two species, because a study by Hardin et al. (Ref. 13) showed no indication that MBT was a teratogen when tested in rats by intraperitoneal injection at the maximum tolerated dose; it was the conclusion of these authors that there was no need for further testing of MBT

In addition, RAPP is trying to locate a Japanese teratogenicity study. In consideration of the existing data, RAPP urges that the Agency limit the developmental toxicity testing to only one species.

As stated in the proposed rule (50 FR 46121), the Agency has reviewed several teratology and reproduction studies (Refs. 13 through 17) and has found them to be inadequate to reasonably predict the developmental and reproductive toxicity of MBT. Several of these studies were designed as screening studies; others were abstracted from Russian literature and details necessary for a thorough review were not available. The Hardin et al. study is of limited value because it was designed as a screening study, and the group sizes used were too small. It has been a longstanding OTS policy to require testing in at least two mammalian species in order to *adequately* assess the potential developmental toxicity of an agent. For these reasons, EPA believes that existing data are insufficient for evaluating the teratogenic risk potential of MBT, and that testing in two species is necessary for the development of adequate data.

3. *Reproductive toxicity testing.* RAPP states that the multigeneration reproductive study cannot be justified by the available data, and points out that, in the proposed test rule for cumene (50 FR 46104; November 6, 1985), the Agency states that a multigeneration reproductive test will not be required for cumene in the absence of evidence of reproductive organ toxicity in a subchronic test. RAPP has reviewed the 90-day subchronic study conducted for NTP (Ref. 18) and submitted in 1985 by Mobay Chemical Corp. No evidence of significant lesions in any of the reproductive organs of rats or mice were reported in that study. RAPP suggests that the Agency postpone a decision on a multigeneration study until after the results of the NTP 2-year chronic bioassay are reviewed and the results of the required dominant lethal assay are available.

When section 4 findings are based on the potential for substantial exposure, as for MBT, EPA requires testing for reproductive effects unless sufficient data adequately describing reproductive effects are available. With regard to the NTP 90-day subchronic study referred to by RAPP, the type of 90-day subchronic study conducted with MBT is not the same as that conducted with cumene. The subchronic study proposed by the Agency for cumene contained specific requirements for extensive evaluation of the reproductive organs of both male and female animals. The subchronic study of MBT conducted for NTP did not contain such an extensive evaluation of reproductive organs since the purpose of this study was to define the appropriate dose levels for a chronic bioassay. Furthermore, histopathology of the reproductive organs will not provide the needed information regarding the integrity of the functioning of the reproductive system. In cases of substantial exposure to an agent, as with MBT, EPA requires data both on morphology and on physiology in the form of a two-generation reproductive effects study. The design of a dominant lethal assay in which only male animals are exposed and only for a relatively short period prior to mating does not provide enough information to adequately describe the effects on reproduction of long-term exposure to a compound. Furthermore, in a dominant lethal study only the male animal is exposed to the test agent and the end points that are assessed are only a small fraction of what is needed to adequately evaluate the potential reproductive toxicity of an agent.

4. *Neurotoxicity testing.* RAPP contends that there is no indication that MBT is a potential neurotoxic agent, and that this is supported by the lack of any histologically observed nervous system damage in the NTP subchronic study, and the lack of structural similarity between MBT and any known neurotoxic solvents. RAPP also believes that no laboratories in this country are available to perform the test, and that the lack of experience with this test would make assessing the results regarding human health difficult. RAPP suggests that if neurotoxicity tests are required, the functional observational battery and neuropathology studies should be conducted in sequence on the same animals.

The NTP bioassay was designed to assess carcinogenicity, and not neurotoxicity. As stated in the proposed rule, no data on the neurotoxic effects of MBT have been found in the literature. Because EPA finds that there is a potential for substantial exposure to MBT, EPA is requiring that adequate neurotoxicity data be developed. With regard to laboratories available to perform the test, EPA points out that the required test protocol is in current use in the research community and on the C_9 aromatic hydrocarbon fraction (50 FR 20675; May 17, 1985), and that industry has conducted such neurotoxicity studies on acrylamide. In addition, EPA has reviewed the availability of contract laboratory facilities to conduct the neurotoxicity testing requirements (Ref. 29) and believes that facilities will be made available for conducting these tests. The Agency does agree that the neuropathology study could be combined with other neurological test protocols without adversely affecting the quality of either study, if the provisions of both guidelines are followed.

5. *Mutagenicity testing.* CMA agrees that the dominant lethal study conducted by Aleksandrov (Ref. 15) is inadequate and that the dominant lethal effect should be further examined by performing a second assay, but states that proceeding directly to the dominant lethal test should preclude the necessity of lower tier testing.

The Agency agrees that the necessity of lower-tier testing for chromosomal aberrations is precluded by industry's agreement to conduct a dominant lethal assay, and by the fact that a 2-year chronic bioassay has already been completed by NTP. Therefore, lower-tier mutagenicity testing will not be required in this rule.

D. New Information

Results of five pharmacokinetic studies (Refs. 21 through 25), comparing the kinetics and metabolism of the oral and dermal routes of exposure, were voluntarily submitted to EPA by CMA. EPA has reviewed these studies and finds them to be well-designed and well-performed. Further pharmacokinetic testing is not being required for MBT at this time; however, additional pharmacokinetic testing may be required at a later date, pending review of the data generated as a result of this final rule.

E. Persons Required to Test

CMA commented that "any testing to be mandated through a test rule should include among those responsible for the testing program persons who import rubber articles containing MBT." CMA pointed out that MBT is manufactured abroad and imported to the United States both as a pure chemical and in mixtures, and as a constituent of articles.

The Agency agrees with this comment, and has clarified in Unit III. E. of this preamble that the term "manufacturers" includes not only importers of MBT itself, but also importers of rubber articles that contain MBT. EPA believes that use and disposal of these articles contribute to human and/or environmental exposures that are part of the basis for the Agency's finding that testing is warranted and necessary.

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Manufacturers are defined by TSCA section 3(7) to include those who "import into the customs territory of the United States." In other TSCA actions under sections 6 and 8, and in discussions of authority under sections 5 and 13, EPA has determined that importation, whether it be in the form of imports of pure chemical substances, mixtures or articles, is included within the TSCA definition of "manufacture."

III. Findings

A. Environmental Effects and Chemical Fate

EPA is basing its final environmental effects and chemical fate testing requirements for MBT on the authority of sections 4(a)(1) (A) and (B) of TSCA. In addition to the information presented in this final rule, the TSCA sections 4(a)(1) (A) and (B) findings are also supported by additional information discussed in the preamble to the proposed test rule for MBT and which is contained in the rulemaking record for this action.

Under TSCA section 4(a)(1)(B)(i), EPA finds that MBT is produced in substantial quantities. This finding takes into account TSCA section 8(a) information that was submitted by the manufacturers of MBT, the indirect production of MBT as a result of the breakdown of MBT-derived accelerators during vulcanization (Ref. 3), and the 1983 sales volume of MBT, which was reported by the U.S. International Trade Commission to be 5,958,000 pounds (Ref. 2).

EPA also finds that there may be substantial quantities of MBT entering the environment. This finding considers TSCA section 8(a) release data submitted by the manufacturers of MBT, releases from processing, disposal, and coolants, and EPA's estimate that over 1 million pounds of MBT may be lost to the environment annually through both direct and indirect discharges. MBT release is also expected to occur as a result of the breakdown of MBT-derived accelerators in discarded rubber products.

Under TSCA section 4(a)(1)(A)(i), EPA finds that the manufacture, processing, use, and disposal of MBT may present an unreasonable risk of injury to organisms in the aquatic environment. EPA is basing this finding on EC50 or LC50 values that are less than 100 times the minimum, median, or maximum predicted environmental concentration (PEC) values, two LC50 values that are less than 1 mg/L, and for an LC50 value greater than 1 mg/L but less than 100 mg/L, the 24-hr to 96-hr LC50 ratio is greater than 2. EPA believes that chronic effects may occur at anticipated environmental concentrations.

EPA has found no data on the chronic effects of MBT on fish and aquatic invertebrates. EPA also concludes that data are insufficient to reasonably predict the biodegradation, indirect photolysis, and chemical mobility of MBT once it is released into the environment. Therefore, under TSCA sections 4(a)(1)(A)(ii) and 4(a)(1)(B)(ii), EPA concludes that available data are insufficient to reasonably determine or predict the chronic effects on fish and aquatic invertebrates from the manufacture, processing, use, and disposal of MBT, or the persistence and mobility of MBT released from such activities. EPA finds that testing of MBT is necessary to develop such data, and believes that data resulting from environmental effects and chemical fate testing will be relevant to a determination as to whether the manufacture, processing, use, or disposal of MBT does or does not present an unreasonable risk of injury to the environment.

The Agency finds that sufficient data are available in the published literature to satisfy the ITC's recommendation that the dissociation constant be determined. Two experimentally-derived values have been found in the literature indicating that the dissociation constant is 6.93 (Ref. 19).

After reviewing and evaluating the existing aquatic toxicity data for MBT, EPA has determined that there are sufficient data available to reasonably predict the acute toxicity of MBT to fish, aquatic invertebrates, and plants. MBT has been shown to exert a high acute toxicity in rainbow trout (Ref. 6) with a 96-hour LC_{50} of 0.75 mg/L. *Daphnia magna* has been shown to have a 48-hour LC_{50} value of 4.1 mg/L (Ref. 9), and *Selenastrum capricornutum* has a 96-hour EC_{50} of 0.23 mg/L (Ref. 11). Therefore, EPA is not requiring any additional acute toxicity tests at this time. Should the existing data and the chronic testing required in this rule provide results indicating a high priority for control of aquatic concentrations of MBT under the Clean Water Act, EPA may at that time propose additional acute and/or chronic testing to establish water quality criteria pursuant to section 304(a)(1) of the Clean Water Act.

The Agency has no evidence of substantial exposure of terrestrial plants along the roadside to MBT from tire dust; therefore, the Agency did not propose, and at this time is not requiring, any acute or chronic toxicity testing for terrestrial plants, as had been recommended by the ITC.

B. Human Health Effects

EPA is basing its final health effects testing requirements for MBT on the authority of TSCA section 4(a)(1)(B). EPA finds that MBT is produced in substantial quantities (See Unit III.A. of this preamble). EPA also finds that there may be substantial human exposure to MBT. The National Occupational Hazard Survey (NOHS), conducted from 1972 to 1974 (Ref. 20), estimates that as many as 558,893 people in the chemical industry may be exposed to MBT. The National Occupational Exposure Survey (NOES) data base (Ref. 4) estimates that 2,398 workers (of whom 119 are female) are exposed to MBT as a result of its presence in finished rubber products.

EPA finds that there are sufficient data availably to reasonable determine or predict the pharmacokinetics, acute effects, chronic effects, oncogenic effects, and gene mutation effects of exposure to MBT at this time. Under TSCA section 4(A)(1)(B)(ii), EPA finds that there are insufficient data available to reasonably determine or predict the effects of the manufacture, processing, use, and disposal of MBT in the areas of developmental toxicity, reproductive toxicity, chromosomal aberrations, and neurotoxicity. EPA finds that testing of MBT is necessary to develop such data, and believes that data resulting from health effects testing will be relevant to a determination as to whether the manufacture, processing, use, or disposal of MBT does or does not present an unreasonable risk of injury to human health.

C. Required Testing and Test Standards

On the basis of these findings, EPA is requiring that chemical fate, environmental effects, and health effects testing be conducted for MBT in accordance with specific test guidelines set forth in 40 CFR Parts 796, 797, and 798. The tests are to be conducted in accordance with EPA's TSCA Good Laboratory Practice Standards in 40 CFR Part 792.

On the basis of the findings presented above for chemical fate testing, the Agency is requiring that MBT be tested for: (1) Biodegradation using the test guideline specified in 40 CFR 796.3160; (2) indirect photolysis screening using the test guideline specified in 40 CFR 795.70,[1] promulgated with this final rule;

[1] § 795.70 *Indirect photolysis screening test: Sunlight photolysis in waters containing dissolved humic substances*, was proposed as § 796.3765 in 51 FR 472; January 6, 1986.

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and (3) chemical mobility using the test guideline specified in 40 CFR 796.2750.

On the basis of the findings presented in Unit III.A. for environmental effects testing, the Agency is requiring that chronic toxicity testing of MBT be conducted on (1) rainbow trout (*Salmo gairdneri*) using the test guideline specified in 40 CFR 797.1600; and (2) *Daphnia magna* using the test guidelines specified in 40 CFR 797.1330.

On the basis of the findings presented in Unit III.B. of this preamble for health effects testing, the Agency is requiring that MBT be tested for: (1) Developmental toxicity in two mammalian species using the test guideline specified in 40 CFR 798.4900; (2) reproductive toxicity using the test guideline specified in 40 CFR 798.4700; (3) neurotoxicity using the test guidelines specified in 40 CFR 798.6050, 798.6200 and 798.6400; and (4) mutagenicity (dominant-lethal assay) using the guidelines specified in 40 CFR 798.5450. A positive result in the dominant-lethal assay may, after a public program review, trigger a heritable translocation assay using the procedure specified in 40 CFR 798.5460. If the dominant-lethal assay is negative, no further chromosomal aberration testing shall be required for MBT.

If the results of the dominant-lethal assay are positive, EPA will hold a public program review prior to requiring the initiation of the heritable translocation assay. Public participation in this program review will be in the form of written comments or a public meeting. Request for public comments or notification of a public meeting will be published in the **Federal Register**. Should EPA determine, from the available weight of evidence, that proceeding to the heritable translocation test is no longer warranted, the Agency would propose to repeal that test requirement and, after public comment, issue a final amendment to rescind the requirement.

EPA is requiring that the TSCA Chemical Fate, Environmental Effects, and Health Effects Test Guidelines referenced in Unit III.C. of this preamble, and revisions, shall be the test standards for the purposes of the required tests for MBT. The TSCA test guidelines for chemical fate, aquatic toxicity, and health effects testing specify generally accepted minimum conditions for determining chemical fate, aquatic organism toxicities, and health effects for substances such as MBT to which humans and the environment are expected to be exposed. The Agency believes that these test methods reflect the current state of the science for testing chemicals such as MBT for the specified end points. The guidelines for rainbow trout and *Daphnia magna* chronic toxicity have been modified in this rule, due to concern over the stability of MBT in water.

D. Test Substance

EPA is requiring that MBT of at least 98 percent purity shall be used as the test substance. MBT of such purity is commercially available.

E. Persons Required to Test

Section 4(b)(3)(B) specifies that the activities for which the Agency makes section 4(a) findings (manufacture, processing, distribution in commerce, use, and/or disposal) determine who bears the responsibility for testing a chemical. Manufacturers and persons who intend to manufacture the chemical are required to test if the findings are based on manufacturing ("manufacture" is defined in section 3(7) of TSCA to include "import," and in this case includes importers of rubber articles that contain MBT). "Manufacture" also includes byproduct manufacture, and while EPA has not identified any byproduct manufacturers of MBT, such persons are subject to the requirements of this test rule. Processors and persons who intend to process the chemical are required to test if the findings are based on processing. Manufacturers and processors and persons who intend to manufacture or process the chemical are required to test if the exposures giving rise to the potential risk occur during distribution in commerce, use, or disposal of the chemical.

Because EPA has found that manufacturing, processing, use, and disposal of MBT results in exposure that may lead to an unreasonable risk, EPA is requiring that persons who manufacture or process, or who intend to manufacture or process, MBT, other than as an impurity, at any time from the effective date of the final test rule to the end of the reimbursement period are subject to the testing requirements contained in this final rule. The end of the reimbursement period will be 5 years after the last final report is submitted or an amount of time equal to that which was required to develop data, whichever is later.

Because TSCA contains provisions to avoid duplicative testing, not every person subject to this rule must individually conduct testing. Section 4(b)(3)(A) of TSCA provides that EPA may permit two or more manufacturers or processors who are subject to the rule to designate one such person or a qualified third person to conduct the tests and submit data on their behalf. Section 4(c) provides that any person required to test may apply to EPA for an exemption from the requirement. EPA has promulgated procedures for applying for TSCA section 4(c) exemptions in 40 CFR Part 790.

Manufacturers (including importers) subject to this rule are required to submit either a letter of intent to perform testing or an exemption application within 30 days after the effective date of the final test rule. The required procedures for submitting such letters and applications are described in 40 CFR Part 790.

Processors subject to this rule, unless they are also manufacturers, will not be required to submit letters of intent or exemption applications, or to conduct testing, unless manufacturers fail to submit notices of intent to test or later fail to sponsor the required tests. The Agency expects that the manufacturers will pass an appropriate portion of the costs of testing on to processors through the pricing of their products or other reimbursement mechanisms. If manufacturers perform all the required tests, processors will be granted exemptions automatically. If manufacturers fail to submit notices of intent to test, or fail to sponsor all the required tests, EPA will publish a separate notice in the **Federal Register** to notify processors to respond; this procedure is described in 40 CFR Part 790.

EPA is not requiring the submission of equivalence data as a condition for exemption from the required testing for MBT. EPA is interested in evaluating the effects attributable to MBT and has specified a relatively pure substance for testing (See Unit III.D. of this preamble).

Manufacturers and processors subject to this test rule must comply with the test rule development and exemption procedures in 40 CFR Part 790 for single-phase rulemaking.

F. Reporting Requirements

EPA is requiring that all data developed under this rule be reported in accordance with its TSCA Good Laboratory Practice (GLP) standards, which appear in 40 CFR Part 792.

In accordance with 40 CFR Part 790 under single-phase rulemaking procedures, test sponsors are required to submit individual study plans at least 45 days before initiation of each test.

EPA is required by TSCA section 4(b)(1)(C) to specify the time period during which persons subject to a test rule must submit test data. Final testing requirements, test standards, and reporting requirements for this MBT test rule are summarized in the following Table.

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REQUIRED TESTING, TEST STANDARDS, AND REPORTING REQUIREMENTS FOR MBT

Test	Test standard (40 CFR citation) (section)	Reporting deadline for final report [1]	Interim (6 month) reports required
Chemical fate:			
1. Aerobic aquatic biodegradation	796.3100	12	1
2. Indirect photolysis—screening	795.70	12	1
3. Sediment and soil adsorption isotherm	796.2750	12	1
Environmental effects:			
1. Fish early life stage toxicity (rainbow trout)	797.1600	12	1
2. Daphnid chronic toxicity	797.1330	12	1
Health effects:			
1. Developmental toxicity (oral)	798.4900	12	1
2. Reproduction and fertility effects (oral)	798.4700	29	4
3. Functional observational battery (oral)	798.6050	12	1
4. Motor activity (oral)	798.6200	12	1
5. Neuropathology (oral)	798.6400	12	1
6. Dominant lethal assay	798.5450	12	1
7. Heritable translocation assay	798.5460	24 [2]	3

[1] Number of months after the effective date of the final rule, except as indicated.

[2] Figure indicates the reporting deadline, in months, calculated from the date of notification of the test sponsor by certified letter or Federal Register notice that, following public program review of all the then existing data for MBT, the Agency has determined that the required testing must be performed.

1. The biodegradation, photolysis, chemical mobility, developmental toxicity, neurotoxicity, and chronic aquatic vertebrate and invertebrate toxicity tests shall be completed and the final results submitted to the Agency within 12 months of the effective date of this final test rule. An interim progress report shall be provided to the Agency 6 months after the effective date of this rule.

2. The reproductive toxicity testing shall be completed and the final results submitted to the Agency within 29 months of the effective date of this final test rule. Interim progress reports shall be provided to the Agency at 6 month intervals after the effective date of this rule, until the final report is submitted to EPA.

3. The dominant-lethal assay and heritable translocation tests for MBT shall be completed and the final results submitted to the Agency after the effective date of this final test rule as follows: Dominant-lethal assay, 12 months; heritable translocation assay, 24 months after notification that testing shall be initiated. There will be a public program review before the heritable translocation test is conducted. Interim progress reports shall be provided to the Agency at 6 month intervals after the effective date of this rule, until the final report is submitted to EPA.

TSCA section 14(b) governs Agency disclosure of all test data submitted pursuant to section 4 of TSCA. Upon receipt of data required by this rule, the Agency will publish a notice of receipt in the Federal Register as required by section 4(d).

Persons who export a chemical which is subject to a section 4 test rule are subject to the export reporting requirements of section 12(b) of TSCA. Final regulations interpreting the requirements of section 12(b) are in 40 CFR Part 707. In brief, as of the effective date of this test rule, an exporter of MBT must report to EPA the first annual export or intended export of MBT to each country. EPA will notify the foreign country concerning the test rule for the chemical.

G. Enforcement Provisions

EPA considers failure to comply with any aspect of a section 4 rule to be a violation of section 15 of TSCA. Section 15(1) of TSCA makes it unlawful for any person to fail or refuse to comply with any rule or order issued under section 4. Section 15(3) of TSCA makes it unlawful for any person to fail or refuse to: (1) Establish or maintain records, (2) submit reports, notices, or other information, or (3) permit access to or copying of records required by the Act or any regulation or rule issued under TSCA.

Additionally, TSCA section 15(4) makes it unlawful for any person to fail or refuse to permit entry or inspection as required by TSCA section 11. Section 11 applies to any "establishment, facility, or other premises in which chemical substances or mixtures are manufactured, processed, stored, or held before or after their distribution in commerce * * *." EPA considers a testing facility to be a place where the chemical is held or stored and, therefore, subject to inspection. Laboratory inspections and data audits will be conducted periodically in accordance with the authority and procedures outlined in TSCA section 11 by designated representatives of the EPA for the purpose of determining compliance with the final rule for MBT. These inspections may be conducted for purposes which include verification that testing has begun, schedules are being met, and reports accurately reflect the underlying raw data, interpretations, and evaluation, and to determine compliance with TSCA GLP standards and the test standards established in this rule.

EPA's authority to inspect a testing facility also derives from section 4(b)(1) of TSCA which directs EPA to promulgate standards for the development of test data. These standards are defined in section 3(12)(B) of TSCA to include those requirements necessary to assure that data developed under testing rules are reliable and adequate, and such other requirements as are necessary to provide such assurance. The Agency maintains that laboratory inspections are necessary to provide this assurance.

Violators of TSCA are subject to criminal and civil liability. Persons who submit materially misleading or false information in connection with the requirement of any provision of this rule may be subject to penalties which may be calculated as if they never submitted their data. Under the penalty provisions of section 16 of TSCA, any person who violates section 15 of TSCA could be subject to a civil penalty of up to $25,000 for each violation, with each day of operation constituting a separate violation. This provision would be applicable primarily to manufacturers that fail to submit a letter of intent or an exemption request and that continue manufacturing after the deadlines for such submissions.

This provision would also apply to processors that fail to submit a letter of intent or an exemption application and continue processing after EPA has notified them of their obligation to submit such documents (see 40 CFR 790.48(b)). Knowing or willful violations could lead to the imposition of criminal penalties of up to $25,000 for each day of violation and imprisonment for up to 1 year. In determining the amount of penalty, EPA will take into account the seriousness of the violation and the degree of culpability of the violator as well as all the other factors listed in TSCA section 16. Other remedies are available to EPA under section 17 of TSCA, such as seeking an injunction to restrain violations of TSCA section 4.

Individuals as well as corporations could be subject to enforcement actions. Sections 15 and 16 of TSCA apply to "any person" who violates provisions of TSCA. EPA may, at its discretion, proceed against individuals as well as companies themselves. In particular, this includes individuals who report

false information or who cause it to be reported. In addition, the submission of false, fictitious, or fraudulent statements is a violation under 18 U.S.C. 1001.

IV. Economic Analysis of Final Rule

To assess the potential economic impact of this rule, EPA has prepared an economic analysis (contained in the public record for this rule) that evaluates the potential for significant economic impact on the industry as a result of the required testing. The economic analysis estimates the costs of conducting the required testing and evaluates the potential for significant adverse economic impact as a result of these test costs by examining four market characteristics of MBT: (1) Price sensitivity of demand, (2) market expectations, (3) industry cost characteristics, and (4) industry structure.

Total testing costs for the required testing for MBT are estimated to range from $434,970 to $583,730. In order to predict the financial decision-making practice of manufacturing firms, these costs have been annualized. Annualized costs are compared with annual revenue as an indication of potential impact. The annualized costs represent equivalent constant costs which would have to be recouped each year of the payback period in order to finance the testing expenditure in the first year.

The annualized test costs (using a 7 percent cost of capital over a period of 15 years) range from $47,758 to $64,088. Based on 1984 production of 47.3 million pounds, the unit test costs range from 0.10 to 0.14 dollar per pound. These costs are equivalent to 0.09 to 0.11 percent of price of the current price of 1.25 dollar per pound.

EPA believes that the potential for adverse economic impact resulting from the costs of testing is low. This conclusion is based on the following observations:

1. The annualized cost of testing is very low, at approximately 0.11 percent of product price in the upper-bound case.

2. Demand for MBT does not appear to be sensitive to a price increase in this range.

Refer to the economic analysis contained in the public record for this rulemaking for a complete discussion of test cost estimation and potential for economic impact resulting from these costs.

V. Availability of Test Facilities and Personnel

Section 4(b)(1) of TSCA requires EPA to consider "* * * the reasonably foreseeable availability of the facilities and personnel needed to perform the testing required under the rule." Therefore, EPA conducted a study to assess the availability of test facilities and personnel to handle the additional demand for testing services created by section 4 test rules. Copies of the study, Chemical Testing Industry: Profile of Toxicological Testing, can be obtained through the National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161 (PB 82-140773). On the basis of this study, EPA believes that there will be available test facilities and personnel to perform the testing specified in this rule.

EPA has reviewed the availability of contract laboratory facilities to conduct the neurotoxicity testing requirements (Ref. 29) and believes that facilities will be made available for conducting these tests. The laboratory review indicates that few laboratories are currently conducting these tests according to TSCA test guidelines and TSCA GLP standards. However, the barriers faced by testing laboratories to gear up for these tests are not formidable. Laboratories will need to invest in testing equipment and personnel training, but EPA believes that these investments will be recovered as the neurotoxicity testing program under TSCA section 4 continues. EPA's expectations of laboratory availability were borne out under the testing requirements of C_9 aromatic hydrocarbon fraction test rule (50 FR 20675; May 17, 1985). Pursuant to that rule, the manufacturers were able to contract with a laboratory to conduct the testing according to TSCA test quidelines and TSCA GLP standards.

VI. Rulemaking Record

EPA has established a record for this rulemaking proceeding [docket number OPTS-42073A]. This record includes:

A. Supporting Documentation

(1) Federal Register notices pertaining to this rule consisting of:

(a) Notice containing the ITC designation of MBT to the Priority List and comments on MBT received in response to that notice. (49 FR 46831; November 29, 1984).

(b) Rules requiring TSCA section 8(a) and 8(d) reporting on MBT (49 FR 46739 and 46741; November 28, 1984).

(c) Notice of EPA's proposed test rule on MBT (50 FR 46121; November 6, 1985).

(d) TSCA test guidelines cited as test standards for this rule (40 CFR Parts 796, 797, and 798).

(e) Notice of final rulemaking on data reimbursement (48 FR 31786; July 11, 1983).

(f) Notice of interim final rule on single-phase test rule development and exemption procedures (50 FR 20652; May 17, 1985).

(g) TSCA GLP standards (48 FR 53992; November 29, 1983).

(2) Economic impact analysis of final test rule for MBT.

(3) Communications consisting of:

(a) Written public comments.

(b) Transcript of public meeting.

(c) Summaries of phone conversations.

(4) Reports published and unpublished factual materials, including: Chemical Testing Industry: Profile of Toxicological Testing (October, 1981).

B. References

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(2) USITC (U.S. International Trade Commission). Synthetic Organic Chemicals, United States Production and Sales. Washington, DC: U.S. Government Printing Office. Pub. No. 1588. (1984).

(3) Monsanto Co. Letter to M. Grief, TSCA Interagency Testing Committee, 401 M St. SW., Washington, DC 20460. (July 16, 1982).

(4) NIOSH (National Institute for Occupational Safety and Health). National occupational exposure survey. U.S. Department of Health, Education, and Welfare. Cincinnati, OH. (1980).

(5) U.S. Environmental Protection Agency (USEPA). Release of mercaptobenzothiazole. Memorandum from EPA-OTS-Design and Development Branch, Office of Toxic Substances (OTS), to Test Rules Development Branch, OTS. (1985).

(6) SRI International. Time-independent toxicity study on Thiotax using rainbow trout as the test organism. SRI Project LSC-1741. Submitted to Monsanto Industrial Chemical Co. (1981).

(7) Monsanto Co. TSCA section 8(d) submission 878215051. Acute (96-hr) toxicity of Thiotax to rainbow trout and bluegill. 1976. USEPA. Office of Toxic Substances. (1985).

(8) Analytical Bio Chemistry Laboratories. Acute toxicity of Thiotax (AB-79-1384365-1a) to fathead minnows (*Pimephales promelas*). Submitted to Monsanto Chemical Co. (August 27, 1979).

(9) Analytical Bio Chemistry Laboratories. Acute toxicity of Thiotax (AB-79-1384365-1d) to *Daphnia magna*. Submitted to Monsanto Chemical Co. (May 31, 1979).

(10) Analytical Bio Chemistry Laboratories. Acute toxicity of NaMBT 50 percent (AB-78-1384320-3a) to *Daphnia magna*. Submitted to Monsanto Chemical Co. (September 30, 1978).

(11) EG&G Bionomics. Toxicity of Thiotax (BN-79-1384365-1e) to the freshwater alga *Selenastrum capricornutum*. Project Number 1497-500. Submitted to Monsanto Chemical Co. (July, 1979).

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freshwater alga *Selenastrum capricornutum*. Project Number H74–500. Submitted to Monsanto Chemical Co. (September, 1978).

(13) Hardin, B.D., Bond, G.P., Sikov, M.R. "Testing of selected work chemicals for teratogenic potential." *Scandanavian Journal of Work, Environment and Health.* (Finland) 7 (Suppl. 4):66–75. (1981).

(14) Bionetics Research Labs. Evaluation of carcinogenic, teratogenic and mutagenic activities of selected pesticides and industrial chemicals. Vol. 2. Teratogenic study in mice and rats. Prepared by BRL Inc. for NCI. NTIS Publication No. 223–160. (1968).

(15) Aleksandrov, S.E. "Effect of vulcanization accelerators on embryonic mortality in rats." *Bulletin of Experimental Biology and Medicine.* 93:87–88. (1982).

(16) Korhonen, A., Hemminki, K., Vaino, H. "Embryotoxicity of benzothiazoles, benzenesulfohydrazide and dithiodimorpholine to the chicken embryo." *Archives of Environmental Contamination and Toxicology.* 11:735–759. (1983).

(17) Korhonen, A., Hemminki, K., Vaino, H. "Toxicity of rubber chemicals toward 3-day chicken embryos." *Scandinavian Journal of Work, Environment and Health.* 9:115–119. (1983).

(18) Physiological Research Laboratories. Subchronic Test Report for Mercaptobenzothiazole. Basic Ordering Agreement 78–60–106002. Performed for the National Toxicology Program. (1981).

(19) Danehy, J.P., Parameswaran, K.N. Acidic dissociation constants of thiols. *Journal of Chemical and Engineering Data.* 13:386–398. (1983).

(20) NIOSH. National occupational hazard survey. U.S. Department of Health, Education, and Welfare. Cincinnati, OH. (1980).

(21) Chemical Manufacturers Association (CMA). "Disposition of 2-mercaptobenzothiazole-ring-UL-^{14}C and 2-mercaptobenzothiazole disulfide-ring-UL-^{14}C in Fischer 344 male and female rats dosed orally." (July 23, 1986).

(22) CMA. "Disposition of 2-mercaptobenzothiazole-ring-UL-^{14}C and 2-mercaptobenzothiazole disulfide-ring-UL-^{14}C in Fischer 344 male and female rats dosed intravenously." (October 21, 1986).

(23) CMA. "Disposition of 2-mercaptobenzothiazole-ring-UL-^{14}C in Fischer 344 male and female rats dosed orally after repeated dosing with the unlabeled compound." (March 20, 1987).

(24) CMA. "Washing efficacy in removal of 2-mercaptobenzothiazole-ring-UL-^{14}C and 2-mercaptobenzothiazole disulfide-ring-UL-^{14}C in Fischer 344 male and female rats and female guinea pigs dosed topically." (March 20, 1987).

(25) CMA. "Absorption and disposition of 2-mercaptobenzothiazole-ring-UL-^{14}C and 2-mercaptobenzothiazole disulfide-ring-UL-^{14}C in Fischer 344 male and female rats dosed topically." (June 16, 1987).

(26) Jungclaus, G.A., Games, L.M. and Hites, R.A. "Identification of trace organic compounds in tire manfacturing plant wastewater." *Analytical Chemistry* 48:1894–1896. (1976).

(27) USEPA. "Industrial Process Profiles for Environmental Use." Chapter 9. The Synthetic Rubber Industry. Cincinnati, OH. EPA 600/2–77–0231. (1977).

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(29) Mathtech, Inc. "Evaluation of TSCA guidelines for neurotoxicity testing: Impact of increased testing requirements." Prepared for Regulatory Impacts Branch, Office of Toxic Substances, USEPA. (April 14, 1987).

Confidential Business Information (CBI), while part of the record, is not available for public view. A public version of the record, from which CBI has been deleted, is available for inspection in the TSCA Public Docket Office, Rm. NE–G004, 401 M Street SW., Washington, DC, from 8 a.m. to 4 p.m., Monday through Friday, except legal holidays.

VII. Other Regulatory Requirements

A. Executive Order 12291

Under Executive Order 12291, EPA must judge whether a rule is "major" and therefore subject to the requirement of a Regulatory Impact Analysis. EPA has determined that this test rule is not major because it does not meet any of the criteria set forth in section 1(b) of the Order; i.e., it will not have an annual effect on the economy of at least $100 million, will not cause a major increase in prices, and will not have significant adverse effect on competition or the ability of U.S. enterprises to compete with foreign enterprises.

This rule was submitted to the Office of Management and Budget (OMB) for review as required by Executive Order 12291. Any written comments from OMB to EPA, and any EPA response to those comments, are included in the rulemaking record.

B. Regulatory Flexibility Act

Under the Regulatory Flexibility Act (15 U.S.C. 601 *et seq.*, Pub. L. 96–354, September 19, 1980), EPA is certifying that this test rule will not have a significant impact on a substantial number of small businesses because: (1) They are not likely to perform testing themselves, or to participate in the organization of the testing effort; (2) they will experience only very minor costs, if any, in securing exemption from testing requirements; and (3) they are unlikely to be affected by reimbursement requirements.

C. Paperwork Reduction Act

The information collection requirements contained in this rule have been approved by the Office of Management and Budget (OMB) under the provisions of the Paperwork Reduction Act, 44 U.S.C. 3501 *et seq.* and has assigned OMB control number 2070–0033.

Public reporting burden for this collection of information is estimated to average 535 hours per response, including time for reviewing instructions, search existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information.

Send comments regarding the burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Chief, Information Policy Branch, PM–223, U.S. Environmental Protection Agency, 401 M Street SW., Washington, DC 20460; and to the Office of Information and Regulatory Affairs, Office of Management and Budget, Washington, DC 20503, marked "Attention: Desk Officer for EPA."

List of Subjects in 40 CFR Parts 795 and 799

Chemicals, Environmental protection, Hazardous substances, Testing, Laboratories, Reporting and recordkeeping requirements.

Dated: August 26, 1988.

Susan F. Vogt,

Acting Assistant Administrator for Pesticides and Toxic Substances.

Therefore, 40 CFR, Chapter I, Subchapter R, is amended as follows:

PART 795—[AMENDED]

1. In Part 795:

a. The authority citation continues to read as follows:

Authority: 15 U.S.C. 2603.

b. Section 795.70 is added to Subpart B, to read as follows:

§ 795.70 Indirect photolysis screening test: Sunlight photolysis in waters containing dissolved humic substances.

(a) *Introduction.* (1) Chemicals dissolved in natural waters are subject to two types of photoreaction. In the first case, the chemical of interest absorbs sunlight directly and is transformed to products when unstable excited states of the molecule decompose. In the second case, reaction of dissolved chemical is the result of chemical or electronic excitation transfer from light-absorbing humic species in the natural water. In contrast to direct photolysis, this photoreaction is governed initially by the spectroscopic properties of the natural water.

(2) In general, both indirect and direct processes can proceed simultaneously. Under favorable conditions the measurement of a photoreaction rate

constant in sunlight (K_{pE}) in a natural water body will yield a net value that is the sum of two first-order reaction rate constants for the direct (k_{DE}) and indirect (k_{IE}) pathways which can be expressed by the relationship

Equation 1

$$k_{pE}=k_{DE}+k_{IE}.$$

This relationship is obtained when the reaction volume is optically thin so that a negligible fraction of the incident light is absorbed and is sufficiently dilute in test chemical; thus the direct and indirect photoreaction processes become first-order.

(3) In pure water only, direct photoreaction is possible, although hydrolysis, biotransformation, sorption, and volatilization also can decrease the concentraton of a test chemical. By measuring k_{pE} in a natural water and k_{DE} in pure water, k_{IE} can be calculated.

(4) Two protocols have been written that measure k_{DE} in sunlight or predict k_{DE} in sunlight from laboratory measurements with monochromatic light (USEPA (1984) under paragraph (f)(14) and (15) of this section; Mill et al. (1981) under paragraph (f)(9) of this section; Mill et al. (1982) under paragraph (f)(10) of this section; Mill et al. (1983) under paragraphs (f)(11) of this section). As a preface to the use of the present protocol, it is not necessary to know k_{DE}; it will be determined under conditions that definitively establish whether k_{IE} is significant with respect to k_{DE}.

(5) This protocol provides a cost effective test method for measuring k_{IE} for test chemicals in a natural water (synthetic humic water, SHW) derived from commercial humic material. It describes the preparation and standardization of SHW. To implement the method, a test chemical is exposed to sunlight in round tubes containing SHW and tubes containing pure water for defined periods of time based on a screening test.

(6) To correct for variations in solar irradiance during the reaction period, an actinometer is simultaneously insolated. From these data, an indirect photoreaction rate constant is calculated that is applicable to clear-sky, near-surface, conditions in fresh water bodies.

(7) In contrast to k_{DE}, which, once measured, can be calculated for different seasons and latitudes, k_{IE} only applies to the season and latitude for which it is determined. This condition exists because the solar action spectrum for indirect photoreaction in humic-containing waters is not generally known and would be expected to change for different test chemicals. For this reason, k_{pE}, which contains k_{IE}, is likewise valid only for the experimental data and latitude.

(8) The value of k_{pE} represents an atypical quantity because k_{IE} will change somewhat from water body to water body as the amount and quality of dissolved aquatic humic substances change. Studies have shown, however, that for optically-matched natural waters, these differences are usually within a factor of two (Zepp et al. (1981) under paragraph (f)(17) of this section).

(9) This protocol consists of three separate phases that should be completed in the following order: In Phase 1, SHW is prepared and adjusted; in Phase 2, the test chemical is irradiated in SHW and pure water (PW) to obtain approximate sunlight photoreaction rate constants and to determine whether direct and indirect photoprocesses are important; in Phase 3, the test chemical is again irradiated in PW and SHW. To correct for photobleaching of SHW and also solar irradiance variations, tubes containing SHW and actinometer solutions are exposed simultaneously. From these data k_{pE} is calculated that is the sum of k_{IE} and k_{DE} (Equation 1) (Winterle and Mill (1985) under paragraph (f)(12) of this section).

(b) *Phase 1—Preparation and standardization of synthetic natural water*—(1) *Approach.* (i) Recent studies have demonstrated that natural waters can promote the indirect (or sensitized) photoreaction of dissolved organic chemicals. This reactivity is imparted by dissolved organic material (DOM) in the form of humic substances. These materials absorb sunlight and produce reactive intermediates that include singlet oxygen ($^1O_2$) (Zepp et al. (1977) under paragraph (f)(20) of this section, Zepp et al. (1981) under paragraph (f)(17) of this section, Zepp et al. (1981) under paragraph (f)(18) of this section, Wolff et al. (1981) under paragraph (f)(16) of this section, Haag et al. (1984) under paragraph (f)(6) of this section, Haag et al. (1984) under paragraph (f)(7) of this section); peroxy radicals ($RO_2\cdot$) (Mill et al. (1981) under paragraph (f)(9) of this section; Mill et al. (1983) under paragraph (f)(8) of this section); hydroxyl radicals ($HO\cdot$) (Mill et al. (1981) under paragraph (f)(9) of this section, Draper and Crosby (1981, 1984) under paragraphs (f)(3) and (4) of this section); superoxide anion ($O_2^{-}\cdot$) and hydroperoxy radicals ($HO_2\cdot$). (Cooper and Zika (1983) under paragraph (f)(1) of this section, Draper and Crosby (1983) under paragraph (f)(2) of this section); and triplet excited states of the humic substances (Zepp et al. (1981) under paragraph (f)(17) of this section, Zepp et al. (1985) under paragraph (f)(21) of this section). Synthetic humic waters, prepared by extracting commercial humic or fulvic materials with water, photoreact similarly to natural waters when optically matched (Zepp et al. (1981) under paragraphs (f)(17) and (18) of this section).

(ii) The indirect photoreactivity of a chemical in a natural water will depend on its response to these reactive intermediates, and possibly others yet unknown, as well as the ability of the water to generate such species. This latter feature will vary from water-to-water in an unpredictable way, judged by the complexity of the situation.

(iii) The approach to standardizing a test for indirect photoreactivity is to use a synthetic humic water (SHW) prepared by water-extracting commercial humic material. This material is inexpensive, and available to any laboratory, in contrast to a specific natural water. The SHW can be diluted to a dissolved organic carbon (DOC) content and uv-visible absorbance typical of most surface fresh waters.

(iv) In recent studies it has been found that the reactivity of SHW mixtures depends on pH, and also the history of sunlight exposure (Mill et al. (1983) under paragraph (f)(11) of this section). The SHW solutions initially photobleach with a time-dependent rate constant. As such, an SHW test system has been designed that is buffered to maintain pH and is pre-aged in sunlight to produce, subsequently, a predictable bleaching behavior.

(v) The purpose of Phase 1 is to prepare, pre-age, and dilute SHW to a standard mixture under defined, reproducible conditions.

(2) *Procedure.* (i) Twenty grams of Aldrich humic acid are added to a clean 2-liter Pyrex Erlenmeyer flask. The flask is filled with 2 liters of 0.1 percent NaOH solution. A stir bar is added to the flask, the flask is capped, and the solution is stirred for 1 hour at room temperature. At the end of this time the dark brown supernatant is decanted off and either filtered through coarse filter paper or centrifuged and then filtered through 0.4)m microfilter. The pH is adjusted to 7.0 with dilute H_2SO_4 and filter sterilized through a 0.2)m filter into a rigorously cleaned 2-liter Erlenmeyer flask. This mixture contains roughly 60 ppm DOC and the absorbance (in a 1 cm path length cell) is approximately 1.7 at 313 nm and 0.7 at 370 nm.

(ii) Pre-aging is accomplished by exposing the concentrated solution in the 2-liter flask to direct sunlight for 4 days in early spring or late fall; 3 days in

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late spring, summer, or early fall. At this time the absorbance of the solution is measured at 370 nm, and a dilution factor is calculated to decrease the absorbance to 0.50 in a 1 cm path length cell. If necessary, the pH is re-adjusted to 7.0. Finally, the mixture is brought to exact dilution with a precalculated volume of reagent-grade water to give a final absorbance of 0.500 in a 1-cm path length cell at 370 nm. It is tightly capped and refrigerated.

(iii) This mixture is SHW stock solution. Before use it is diluted 10-fold with 0.010 M phosphate buffer to produce a pH 7.0 mixture with an absorbance of 5.00×10^{-2} at 370 nm, and a dissolved organic carbon of about 5 ppm. Such values are characteristic of many surface fresh waters.

(3) *Rationale.* The foregoing procedure is designed to produce a standard humic-containing solution that is pH controlled, and sufficiently aged that its photobleaching first-order rate constant is not time dependent. It has been demonstrated that after 7 days of winter sunlight exposure, SHW solutions photobleached with a nearly constant rate constant (Mill et al. (1983) under paragraph (f)(11) of this section).

(c) *Phase 2—Screening test*—(1) *Introduction and purpose.* (i) Phase 2 measurements provide approximate solar photolysis rate constants and half-lives of test chemicals in PW and SHW. If the photoreaction rate in SHW is significantly larger than in PW (factor of > 2X) then the test chemical is subject to indirect photoreaction and Phase 3 is necessary. Phase 2 data are needed for more accurate Phase 3 measurements, which require parallel solar irradiation of actinometer and test chemical solutions. The actinometer composition is adjusted according to the results of Phase 2 for each chemical, to equalize as much as possible photoreaction rate constants of chemical in SHW and actinometer.

(ii) In Phase 2, sunlight photoreaction rate constants are measured in round tubes containing SHW and then mathematically corrected to a flat water surface geometry. These rate constants are not corrected to clear-sky conditions.

(2) *Procedure.* (i) Solutions of test chemicals should be prepared using sterile, air-saturated, 0.010 M, pH 7.0 phosphate buffer and reagent-grade (or purer) chemicals.[1] Reaction mixtures should be prepared with chemicals at concentrations at less than one-half their solubility in pure water and at concentrations such that, at any wavelengths above 290 nm, the absorbance in a standard quartz sample cell with a 1-cm path length is less than 0.05. If the chemicals are too insoluble in water to permit reasonable handling or analytical procedures, 1-volume percent acetonitrile may be added to the buffer as a cosolvent.

(ii) This solution should be mixed 9.00:1.00 by volume with PW or SHW stock solution to provide working solutions. In the case of SHW, it gives a ten-fold dilution of SHW stock solution. Six mL aliquots of each working solution should then be transferred to separate 12 × 100 mm quartz tubes with screw tops and tightly sealed with Mininert valves.[2] Twenty four tubes are required for each chemical solution (12 samples and 12 dark controls), to give a total of 48 tubes.

(iii) The sample tubes are mounted in a photolysis rack with the tops facing geographically north and inclined 30° from the horizontal. The rack should be placed outdoors over a black background in a location free of shadows and excessive reflection.

(iv) Reaction progress should be measured with an analytical technique that provides a precision of at least ±5 percent. High pressure liquid chromatography (HPLC) or gas chromatograph (GC) have proven to be the most general and precise analytical techniques.

(v) Sample and control solution concentrations are calculated by averaging analytical measurements for each solution. Control solutions should be analyzed at least twice at zero time and at other times to determine whether any loss of chemical in controls or samples has occurred by some adventitious process during the experiment.

(vi) Whenever possible the following procedures should be completed in clear, warm, weather so that solutions will photolyze more quickly and not freeze.

(A) Starting at noon on day zero, expose to sunlight 24 sample tubes mounted on the rack described above. Tape 24 foil-wrapped controls to the bottom of the rack.

(B) Analyze two sample tubes and two unexposed controls in PW and SHW for chemical at 24 hours. Calculate the round tube photolysis rate constants $(k_p)_{SHW}$ and $(k_p)_W$ if the percent conversions are J 20 percent but F 80 percent. The rate constants $(k_p)_{SHW}$ and $(k_p)_W$ are calculated, respectively, from Equations 2 and 3:

Equation 2

$$(k_p)_{SHW} = (1/t)Pn(C_o/C_t)_{SHW} \text{ (in } d^{-1})$$

Equation 3

$$(k_p)_W = (1/t)Pn(C_o/C_t)_W \text{ (in } d^{-1}),$$

where the subscript identifies a reaction in SHW or PW; t is the photolysis time in calendar days; C_o is the initial molar concentration; and C_t is the molar concentration in the irradiated tube at t. In this case t=1 day.

(C) If less than 20 percent conversion occurs in SHW in 1 day, repeat the procedure for SHW and PW at 2 days, 4 days, 8 days, or 16 days, or until 20 percent conversion is reached. Do not extend the experiment past 16 days. If less than 20 percent photoreaction occurs in SHW at the end of 16 days the chemical is "photoinert". Phase 3 is not applicable.

(D) If more than 80 percent photoreaction occurs at the end of day 1 in SHW, repeat the experiment with eight each of the remaining foil-wrapped PW and SHW controls. Divide these sets into four sample tubes each, leaving four foil-wrapped controls taped to the bottom of the rack.

(*1*) Expose tubes of chemical in SHW and PW to sunlight starting at 0900 hours and remove one tube and one control at 1, 2, 4, and 8 hours. Analyze all tubes the next day.

(*2*) Extimate $(k_p)_{SHW}$ for the first tube in which photoreaction is J 20 percent but F 80 percent. If more than 80 percent conversion occurs in the first SHW tube, report: "The half-life is less than one hour" and end all testing. The chemical is "photolabile." Phase 3 is not applicable.

(*3*) The rate constants $(k_p)_{SHW}$ and $(k_p)_W$ are calculated from equations 2 and 3 but the time of irradiation must be adjusted to reflect the fact that day-averaged rate constants are approximately one-third of rate constants averaged over only 8 daylight hours. For 1 hour of insolation enter t=0.125 day into equation 2. For reaction times of 2, 4, and 8 hours enter 0.25, 0.50 and 1.0 days, respectively. Proceed to Phase 3 testing.

(*4*) Once $(k_p)_{SHW}$ and $(k_p)_W$ are measured, determine the ratio R from equation 4:

Equation 4

$$R = (k_p)_{SHW}/(k_p)_W.$$

The coefficient R, defined by Equation 4, is equal to $[(k_1+k_D)/k_D]$. If R is in the

[1] The water should be ASTM Type IIA, or an equivalent grade.

[2] Mininert Teflon sampling vials are available from Alltech Associates, Inc., 202 Campus Dr., Arlington Heights, IL 60004.

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range 0 to 1, the photoreaction is inhibited by the synthetic humic water and Phase 3 does not apply. If R is in the range 1 to 2, the test chemical is marginally susceptable to indirect photolysis. In this case, Phase 3 studies are optional. If R is greater than 2, Phase 3 measurements are necessary to measure k_{pE} and to evaluate k_{IE}.

(vii) Since the rate of photolysis in tubes is faster than the rate in natural water bodies, values of near-surface photolysis rate constants in natural and pure water bodies, k_{pE} and k_{DE}, respectively, can be obtained from $(k_p)_{SHW}$ and $(k_p)_W$ from Equations 5 and 6:

Equation 5

$$k_{pE}=0.45(k_p)_{SHW}$$

Equation 6

$$k_{DE}=0.45(k_p)_W.$$

The factor 0.45 is an approximate geometric correction for scattered light in tubes versus horizontal surfaces. A rough value of k_{IE}, the rate constant for indirect photolysis in natural waters or SHW, can be estimated from the difference between k_{pE} and k_{DE} using Equation 7:

Equation 7

$$k_{IE}=k_{pE}-k_{DE}.$$

(3) *Criteria for Phase 2.* (i) If no loss of chemical is found in dark control solutions compared with the analysis in tubes at zero time (within experimental error), any loss of chemical in sunlight is assumed to be due to photolysis, and the procedure provides a valid estimate of k_{pE} and k_{DE}. Any loss of chemical in the dark-control solutions may indicate the intervention of some other loss process such as hydrolysis, microbial degradation, or volatilization. In this case, more detailed experiments are needed to trace the problem and if possible eliminate or minimize the source of loss.

(ii) Rate constants determined by the Phase 2 protocol depend upon latitude, season, and weather conditions. Note that $(k_p)_{SHW}$ and k_D values apply to round tubes and k_{pE} and k_{DE} values apply to a natural water body. Because both $(k_p)_{SHW}$ and k_D are measured under the same conditions the ratio $((k_p)_{SHW}/k_D)$ is a valid measure of the susceptibility of a chemical to indirect photolysis. However, since SHW is subject to photobleaching, $(k_p)_{SHW}$ will decrease with time because the indirect rate will diminish. Therefore, $R < 2$ is considered to be a conservative limit because $(k_p)_{SHW}$ will become systematically smaller with time.

(4) *Rationale.* The Phase 2 protocol is a simple procedure for evaluating direct and indirect sunlight photolysis rate constants of a chemical at a specific time of year and latitude. It provides a rough rate constant for the chemical in SHW that is necessary for Phase 3 testing. By comparison with the direct photoreaction rate constant, it can be seen whether the chemical is subject to indirect photoreaction and whether Phase 3 tests are necessary.

(5) *Scope and limitations.* (i) Phase 2 testing separates test chemicals into three convenient categories: "Photolabile", "photoinert", and those chemicals having sunlight half-lives in round tubes in the range of 1 hour to 50 days. Chemicals in the first two categories fall outside the practical limits of the test, and cannot be used in Phase 3. All other chemicals are suitable for Phase 3 testing.

(ii) The test procedure is simple and inexpensive, but does require that the chemical dissolve in water at sufficient concentrations to be measured by some analytical technique but not have appreciable absorbance in the range 290 to 825 nm. Phase 2 tests should be done during a clear-sky period to obtain the best results. Testing will be less accurate for chemicals with half-lives of less than 1 day because dramatic fluctuations in sunlight intensity can arise from transient weather conditions and the difficulty of assigning equivalent reaction times. Normal diurnal variations also affect the photolysis rate constant. Phase 3 tests should be started as soon as possible after the Phase 2 tests to ensure that the $(k_p)_{SHW}$ estimate remains valid.

(6) *Illustrative Example.* (i) Chemical A was dissolved in 0.010 M pH 7.0 buffer. The solution was filtered through a 0.2)m filter, air saturated, and analyzed. It contained 1.7×10^{-5} M A, five-fold less than its water solubility of 8.5×10^{-5} M at 25°C. A uv spectrum (1-cm path length) versus buffer blank showed no absorbance greater than 0.05 in the wavelength interval 290 to 825 nm, a condition required for the Phase 2 protocol. The 180 mL mixture was diluted by the addition of 20 mL of SHW stock solution.

(ii) The SHW solution of A was photolyzed in sealed quartz tubes (12×100 mm) in the fall season starting on October 1. At the end of 1 and 2 days, respectively, the concentration of A was found to be 1.13×10^{-5} M and 0.92×10^{-5} M compared to unchanged dark controls (1.53×10^{-5} M).

(iii) The tube photolysis rate constant of chemical A was calculated from Equation 2 under paragraph (c)(2)(vi)(B) of this section. The first time point at day 1 was used because the fraction of A remaining was in the range 20 to 80 percent:

$$(k_p)_{SHW}=(1/1d)Pn(1.53\times10^{-5}/1.13\times10^{-5})$$
$$(k_p)_{SHW}=0.30\ d^{-1}.$$

(iv) From this value, k_{pE} was found to be 0.14 d^{-1} using equation 5 under paragraph (c)(2)(vii) of this section:

$$k_{pE}=0.45(0.30\ d^{-1})=0.14d^{-1}.$$

(v) From measurements in pure water, k_D for chemical A was found to be 0.085 d^{-1}. Because the ratio of $(k_p)_{SHW}/k_D(=3.5)$ is greater than 2, Phase 3 experiments were started.

(d) *Phase 3—Indirect photoreaction with actinometer: Calculation of* k_{IE} *and* k_{pE}—(1) *Introduction and purpose.*

(i) The purpose of Phase 3 is to measure k_{Io}, the indirect photolysis rate constant in tubes, and then to calculate k_{pE} for the test chemical in a natural water. If the approximate $(k_p)_{SHW}$ determined in Phase 2 is not significantly greater than k_D measured for the experiment date of Phase 2, then Phase 3 is unnecessary because the test chemical is not subject to indirect photoreaction.

(ii) In the case $(k_p)_{SHW}$ is significantly larger than k_D, Phase 3 is necessary. The rate constant $(k_p)_{SHW}$ is used to choose an actinometer composition that matches the actinometer rate to the test chemical rate. Test chemical solutions in SHW and in pure water buffer are then irradiated in sunlight in parallel with actinometer solutions, all in tubes.

(iii) The actinometer used is the *p*-nitroacetophenone-pyridine (PNAP/PYR) system developed by Dulin and Mill (1982) under paragraph (f)(5) of this section and is used in two EPA test guidelines (USEPA (1984) under paragraphs (f) (14) and (15) of this section). By varying the pyridine concentration, the PNAP photolysis half-life can be adjusted over a range of several hours to several weeks. The starting PNAP concentration is held constant.

(iv) SHW is subject to photobleaching that decreases its ability to promote indirect photolysis based on its ability to absorb sunlight. This effect will be significant when the test period exceeds a few days. To correct for photobleaching, tubes containing SHW are irradiated in action to the other tubes above.

(v) At any time, the loss of test chemical is given by Equation 8 assuming actinometric correction to constant light flux:

Equation 8

$$-(d[C]/dt)=k_I[C]+k_D[C].$$

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(vi) The indirect photolysis rate constant, k_i, is actually time dependent because SHW photobleaches; the rate constant k_i, after pre-aging, obeys the formula:

Equation 9

$$k_i = k_{io} \exp(-kt),$$

in which k_{io} is the initial indirect photoreaction rate constant and k is the SHW photobleaching rate constant. After substituting equation 9 for k_i in Equation 8 under paragraph (d)(1)(v) of this section, and rearranging, one obtains

$$-(d[C]/[C] = k_{io}[\exp(-kt)]dt + k_D dt.$$

This expression is integrated to give Equation 10:

Equation 10

$$Pn(C_o/C)_{SHW} = (k_{io}/k)[1-\exp)-kt)] + k_D t.$$

The term (k_{io}/k) can now be evaluated. Since in pure water, $Pn(C_o/C)_W = k_D t$, then subtracting this equation from Equation 10 gives

Equation 11

$$Pn(C_o/C)_{SHW} - Pn(c_o/C)_W = (k_{io}/k)[1-\exp(-kt)].$$

The photobleaching fraction, [1-exp(-kt)], is equivalent to the expression $[1-(A_{370}/A^{\circ}_{370})]$, where A°_{370} and A_{370} are the absorbances at 370 nm, and are proportional to humic sensitizer content at times zero and t. Therefore, (k_{io}/k) is derived from the slope of a linear regression using $[Pn(C_o/C)_{SHW} - Pn(C_o/C)_W]$ as the dependent variable and $[1-(A_{370}/A^{\circ}_{370})_{SHW}]$ as the independent variable.

(vii) To evaluate k_{io}, the parameter k has to be evaluated under standard sunlight conditions. Therefore, the photolysis rate constant for the PNAP/PYR actinometer (k_A) is used to evaluate k by linear regression on Equation 12:

Equation 12

$$Pn(A^{\circ}_{370}/A_{370}) = (k/k_A)Pn(C_o/C)_{PNAP}.$$

where the slope is (k/k_A) and the value of k_A is calculated from the concentration of pyridine and the absorption of light by PNAP: $k_A = 2.2(0.0169)[PYR]k_a$. Values of k_a are listed in the following Table 1.

TABLE 1.—DAY AVERAGED RATE CONSTANT (k_a) [1] FOR SUNLIGHT ABSORPTION BY PNAP AS A FUNCTION OF SEASON AND DECADIC LATITUDE [2]

Latitude	Season			
	Spring	Summer	Fall	Winter
20°N	515	551	409	327
30°N	483	551	333	232
40°N	431	532	245	139
50°N	362	496	154	64

[1] $k_a = @\ e_{\alpha\lambda} L_\lambda$ in the units of day $^{-1}$, (Mill et al. (1982) under paragraph (f)(10) of this section).
[2] For use in Equation 15 under paragraph (d)(2)(i) of this section.

The value of k_{io} is then given by Equation 13:

Equation 13

$$k_{io} = (k_{io}/k)(k/k_A)k_A.$$

(viii) To obtain k_D, determine the ratio (k_D/k_A) from a linear regression of $Pn(C_o/C)_W$ versus $Pn(C_o/C)_{PNAP}$ according to Equation 13a:

Equation 13a

$$Pn(C_o/C)_W = (k_D/k_A)Pn(C_o/C)_{PNAP}.$$

The slope is (k_D/k_A), and k_D is obtained by multiplication of this slope with the known value of k_A: i.e., $k_D = (k_D/k_A)k_A$.

(ix) Then, $(k_p)_{SHW}$ values in SHW are determined by summing k_D and K_{io} as follows:

Equation 14

$$(k_p)_{SHW} = k_{io} + k_D.$$

(x) Finally, $k_{p\pi}$ is calculated from the precise relationship, Equation 5a:

Equation 5a

$$k_{p\pi} = 0.455(k_p)_{SHW}.$$

(2) *Procedure.* (i) Using the test chemical photoreaction rate constant in round tubes, $(k_p)_{SHW}$ determined in Phase 2 under paragraph (c) of this section, and the absorption rate constant, k_a found in Table 1, under paragraph (d)(1)(vii) of this section, calculate the molar pyridine concentration required by the PNAP/PYR actinometer using Equation 15:

Equation 15

$$[PYR]/M = 26.9[(k_p)_{SHW}/k_a].$$

This pyridine concentration makes the actinometer rate constant match the test chemical rate constant.

(A) The variable k_a $(= @\ e_{\alpha\lambda} L_\lambda)$ is equal to the day-averaged rate constant for sunlight absorption by PNAP (USEPA (1984) under paragraph (f)(14) of this section; Mill et al. (1982) under paragraph (f)(10) of this section, Zepp and Cline (1977) under paragraph (f)(19) of this section) which changes with season and latitude.

(B) The variable k_a is selected from Table 1 under paragraph (d)(1)(vii) of this section for the season nearest the mid-experiment date of Phase 2 studies and the decadic latitude nearest the experimental site.

(ii) Once [PYR] is determined, an actinometer solution is prepared by adding 1.00 mL of 1.0×10^{-2} M (0.165 gms/100 mL) PNAP stock solution (in CH_3CN solvent) and the required volume, V, of PYR to a 1 liter volumetric flask. The flask is then filled with distilled water to give 1 liter of solution. The volume V can be calculated from Equation 16:

Equation 16

$$V/mL = [PYR]/0.0124.$$

The PNAP/PYR solutions should be wrapped with aluminum foil and kept out of bright light after preparation.

(iii) The following solutions should be prepared and individually added in 6.000 mL aliquots to 12/100 mm quartz sample tubes; 8 tubes should be filled with each solution:

(A) PNAP/PYR actinometer solution.

(B) Test chemical in pH 7.0, 0.010 M phosphate buffer.

(C) Test chemcial in pH 7.0, 0.010 M phosphate buffer/SHW.

(D) pH 7.0, 0.010 M phosphate buffer/SHW. Four tubes of each set are wrapped in foil and used as controls.

(iv) The tubes are placed in the photolysis rack (Phase 2, Procedure) at 0900 hours on day zero, with the controls taped to the bottom of the rack. One tube of each composition is removed, along with their respective controls, according to a schedule found in Table 2, which categorizes sampling times on the basis of $(k_p)_{SHW}$ determined in Phase 1.

TABLE 2.—CATEGORY AND SAMPLING PROCEDURE FOR TEST AND ACTINOMETRY SOLUTIONS

Category	k_p (d^{-1})$_{SHW}$	Sampling procedure
A	5.5 J K_p J 0.69	Sample at 0, 1, 2, 4, and 8h.
B	0.69<k_p J 0.017	Sample at 0, 1, 2, 4, and 8d.
C	0.17<k_p J 0.043	Sample at 0, 4, 8, 16, and 32d.

(v) The tubes containing PNAP, test chemical, and their controls are analyzed for residual concentrations soon after the end of the experiment. PNAP is conveniently analyzed by HPLC, using a 30 cm C_{18} reverse column and a uv detector set at 280 nm. The

mobile phase is 2 percent acetic acid, 50 percent acetonitrile and 48 percent water (2 mL/min flow rate). Tubes containing only SHW (solution D) should be analyzed by absorption spectroscopy at 370 nm after storage at 4°C in the dark. The absorbance range to be measured is 0.05 to 0.01 AU (1 cm).

(vi) If controls are well-behaved and show no significant loss of chemical or absorbance change, then k_t can be calculated. In tabular form (see Table 4 under paragraph (d)(6)(iii)(A) of this section) arrange the quantities $Pn(C_o/C_t)_{SHW}$, $Pn(C_o/C_t)_W$, $[1-(A_{370}/A°_{370})]$, $Pn(A°_{370}/A_{370})$, and $Pn(C_o/C)_{PNAP}$ in order of increasing time. According to Equation 11 under paragraph (d)(1)(vi) of this section in the form of Equation 17.

Equation 17

$$Pn(C_o/C)_{SHW} - Pn(C_o/C)_W = (k_{Io}/k)[1-(A_{370}/A°_{370})],$$

plot the quantities $[Pn(C_o/C_t)_{SHW} - Pn(C_o/C_t)_W]$ versus the independent variable $[1-(A_{370}/A°_{370})]$. Obtain the slope (S1) by least square linear regression. Under the assumptions of the protocol, $S1=(k_{Io}/k)$.

(vii) According to Equation 12 under paragraph (d)(1)(vii) of this section, plot the quantities $Pn(A°_{370}/A_{370})$ versus the independent variable $Pn(C_o/C_t)_{PNAP}$. Obtain the slope (S2) by least squares linear regression on Equation 12 under paragraph (d)(1)(vii) of this section. Under the assumptions of the protocol, $S2=(k/k_A)$.

(viii) Then, using Equation 13a under paragraph (d)(1)(vii) of this section, determine the slope (S3) by least squares linear regression. Under the assumptions of the protocol, S3 is equal to (k_D/k_A).

(ix) From Equation 18

Equation 18

$$k_A = 0.0372[PYR]k_a,$$

calculate k_A using k_a values found in Table 1 under paragraph (d)(1)(vii) of this section. The value of k_a chosen must correspond to the date closest to the mid-experiment date and latitude closest to that of the experimental site.

(x) The indirect photoreaction rate constant, k_{Io}, is determined using Equation 19,

Equation 19

$$k_{Io} = (S1)(k_A)(S2).$$

by incorporating the quantities k_A, S1, and S2 determined as described in paragraphs (d)(2) (ix), (vi), and (vii) of this section, respectively.

(xi) The rate constant k_D is calculated from Equation 20,

Equation 20

$$k_D = (S3)(k_A),$$

using the quantities S3 and k_A determined as described above.

(xii) Then, $(k_p)_{SHW}$ is obtained by summing k_D and k_{Io}, as described by Equation 14 in paragraph (d)(1)(ix) of this section:

Equation 14

$$(k_p)_{SHW} = k_{Io} + k_D.$$

(xiii) Finally, k_{pE} is obtained by multiplying $(k_p)_{SNW}$ by the factor 0.455, as described by Equation 5a in paragraph (d)(1)(x) of this section:

Equation 5a

$$k_{pE} = 0.455\ (k_p)_{SHW}$$

As determined, k_{pE} is the net environmental photoreaction rate constant. It applies to clear sky conditions and is valid for predicting surface photoreaction rates in an average humic containing freshwater body. It is strictly valid only for the experimental latitude and season.

(3) *Criteria for Phase 3.* As in Phase 2. Phase 3 tests are assumed valid if the dark controls are well behaved and show no significant loss of chemical. In such a case, loss of test chemical in irradiated samples is due to photoreaction.

(4) *Rationale.* Simultaneous irradiation of a test chemical and actinometer provide a means of evaluating sunlight intensities during the reaction period. Parallel irradiation of SHW solutions allows evaluation of the extent of photobleaching and loss of sensitizing ability of the natural water.

(5) *Scope and limitations of Phase 3 protocol.* Test chemicals that are classified as having half-lives in SHW in the range of 1 hour to 50 days in Phase 2 listing are suitable for use in Phase 3 testing. Such chemicals have photoreaction half-lives in a range accommodated by the PNAP/PYR actinometry in sunlight and also accommodate the persistence of SHW in sunlight.

(6) *Illustrative example.* (i) From Phase 2 testing, under paragraph (c)(6)(iii) of this section, chemical A was found to have a photolysis rate constant, $(k_p)_{SHW}$, of 0.30 d^{-1} in fall in round tubes at latitude 33°N. Using Table 1 under paragraph (d)(1)(vii) of this section for 30°N, the nearest decadic latitude, a fall value of k_a equal to 333 d^{-1} is found for PNAP. Substitution of $(k_p)_{SHW}$ and k_a into Equation 15 under paragraph (d)(2)(i) of this section gives [PYR] = 0.0242 M. This is the concentration of pyridine that gives an actinometer rate constant of 0.30 d^{-1} in round tubes in fall at this latitude.

(ii) The actinometer solution was made up by adding a volume of pyridine (1.95 mL) calculated from equation 16 under paragraph (d)(2)(ii) of this section to a 1 liter volumetric flask containing 1.00 mL of 1.00×10^{-2} M PNAP in acetonitrile. The flask was filled to the mark with distilled water to give final concentrations of [PYR] = 0.0242 M and $[PNAP] = 1.00 \times 10^{-5}$ M. Ten tubes of each of the following solutions were placed in the photolysis rack at 1,200 hours on day zero:

(A) Chemical A (1.53×10^{-5}M) in standard SHW (0.010 M, pH 7 phosphate buffer).

(B) Chemical A (1.53×10^{-5}), in 0.010 M, pH 7 phosphate buffer.

(C) SHW standard solution diluted with water 0.90 to 1.00 to match solution A.

(D) PNAP/PYR actinometer solution. Ten additional foil-wrapped controls of each mixture were taped to the bottom of the rack.

(iii) The test chemical had been placed in category B, Table 2 under the paragraph (d)(2)(iv) of this section, on the basis of its Phase 2 rate constant under paragraph (c) of this section. Accordingly, two tubes of each irradiated solution and two tubes of each blank solution were removed at 0, 1, 2, 4, and 8 days at 1,200 hours. The averaged analytical results obtained at the end of the experiment are shown in the following Table 3.

TABLE 3.—CHEMICAL ANALYTICAL RESULTS FOR ILLUSTRATIVE EXAMPLE, PHASE 3

Day	$10^6[C]^{SHW}$, M	$10^6[C]^W$, M	A^{SHW}_{370}	10^6 [PNAP], M
0	1.53	1.53	0.0500	1.00
1	1.03	1.40	0.0470	0.810
2	0.760	1.30	0.0440	0.690
4	0.300	1.01	0.0370	0.380
8	0.130	0.800	0.0320	0.220

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Data for solutions A through D are given in column 2 through 5, respectively. No significant chemical loss was found in the dark controls.

(A) From these items the functions $Pn(C_o/C)_{SNW}$, $Pn(C_o/C)_W$, $[1-(A_{370}/A°_{370})_{SNW}]$, $Pn(A°_{370}/A_{370})$, and $Pn(C_o/C)_{PNAP}$ were calculated, as shown in the following Table 4 which was derived from Table 3 under paragraph (d)(6)(iii) of this section:

TABLE 4.—PHOTOREACTION FUNCTION FOR ILLUSTRATIVE EXAMPLES, PHASE 3, DERIVED FROM TABLE 3

Day	$Pn(C_o/C)_{SHW}$	$Pn(C_o/C)_W$	$1-(A_{370}/A°_{370})$	$Pn(A°_{370}/A_{370})$	$Pn(C_o/C)_{PNAP}$
0	0	0	0	0	0
1	0.396	0.0888	0.0600	0.0618	0.211
2	0.700	0.163	0.120	0.128	0.371
4	1.629	0.415	0.260	0.301	0.968
8	2.465	0.648	0.360	0.446	1.514

(B) Slope $S1=(k_{1o}/k)$ was calculated according to Equation 17 under paragraph (d)(2)(vi) of this section and was found to be 4.96 by a least squares regression with a correlation coefficient equal to 0.9980. The following Figure 1 shows a plot of Equation 17 under paragraph (d)(2)(vi) of this section and its best-fit line.

(C) Slope $S2=(k/k_a)$ was also derived from Table 4 under paragraph (d)(6)(iii)(A) of this section by a fit of $Pn(A_{o370}/A_{370})_{SHW}$ and $Pn(C_o/C)_{PNAP}$ to Equation 12 under paragraph (d)(1)(vii) of this section. This plot is displayed in the following Figure 2; the slope S2 was found to be 0.295 and the correlation coefficient was equal to 0.9986.

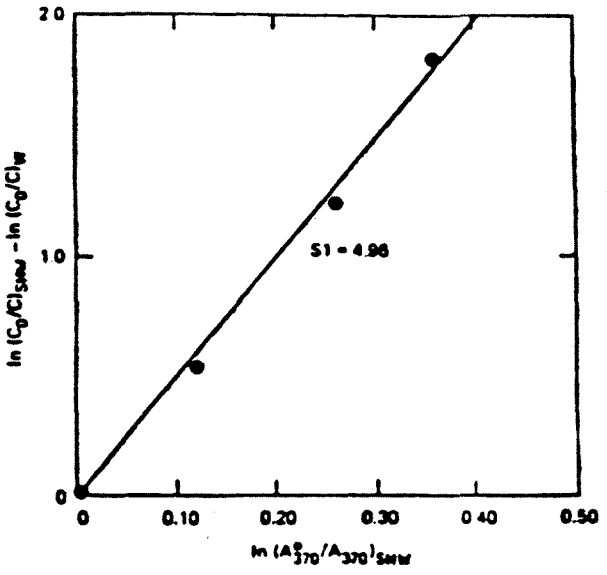

Figure 1.—Graphic determination of $S1=(k_{1o}/k)$ based on Equation 17 under paragraph (d)(2)(vi) of this section.

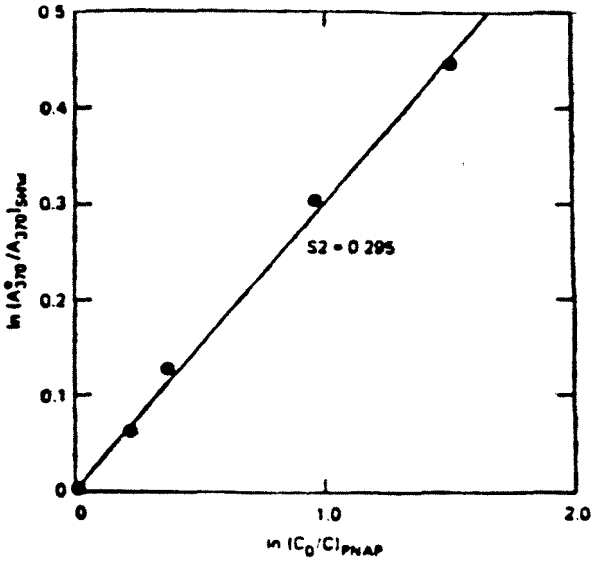

Figure 2.—Graphic determination of $S2=(k/k_A)$ based on Equation 12 under paragraph (d)(1)(vii) of this section.

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(D) Using the data in columns 3 and 6 in Table 4 under paragraph (d)(6)(iii)(A) of this section, slope S3 was calculated by regression from Equation 13a under paragraph (d)(1)(viii) of this section and was found to be 0.428 with correlation coefficient euqal to 0.99997.

(E) Using Equation 18 under paragraph (d)(2)(ix) of this section, k_A was found to be $=0.300\ d^{-1}$.

(F) The values of S1, S2, and k_A were then combined in Equation 19 under paragraph (d)(2)(x) of this section to give k_{lo} as follows:

Equation 19

$$k_{lo}=(4.96)(0.300)(0.295)=0.439\ d^{-1}.$$

(G) The rate constant k_D was calculated from the product of S3 and k_A as expressed in Equation 20 under paragraph (d)(2)(xi) of this section as follows:

Equation 20

$$k_D=(0.428)(0.300)=0.128 d^{-1}.$$

(H) The sum of k_D and k_{lo} was multiplied by 0.455 to obtain k_{pE} as follows:

Equation 21

$$k_{pE}=(0.455)(0.439+0.128)d^{-1}=0.258 d^{-1}.$$

(I) Since k_{pE} is a first-order rate constant, the half-life, $t_{1/2E}$, is given by Equation 22:

Equation 22

$$t_{1/2E}=0.693/k_{pE}.$$

Substituting the value of k_{pE} from Equation 21 under paragraph (d)(6)(iii)(H) of this section in Equation 22 yielded

Equation 23

$$t_{1/2E}=0.693/0.258d^{-1}=2.7d.$$

(e) *Data and reporting*—(1) *Test conditions*—(i) *Specific analytical and recovery procedures.* (A) Provide a detailed description or reference for the analytical procedures used, including the calibration data and precision.

(B) If extraction methods were used to separate the solute from the aqueous solution, provide a description of the extraction method as well as the recovery data.

(ii) *Other test conditions.* (A) Report the site and latitude where the photolysis experiments were carried out.

(B) Report the dates of photolysis, weather conditions, times of exposure, and the duration of exposure.

(C) If acetonitrile was used to solubilize the test chemical, report the volume percent.

(D) If a significant loss of test chemical occurred in the control solutions for pure water and SHW, indicate the causes and how they were eliminated or minimized.

(2) *Test data report*—(i) Phase 2 Screening Test under paragraph (c) of this section. (A) Report the initial molar concentration of test chemical, C_o, in pure water and SHW for each replicate and the mean value.

(B) Report the molar concentration of test chemical, C_t, in pure water and SHW for each replicate and the mean value for each time point t.

(C) Report the molar concentration of test chemical for each replicate control sample and the mean value for each time point.

(D) Report the values of $(k_p)_{SHW}$ and $(k_p)_W$ for the time point t in which the fraction of test chemical photoreacted is in the range 20 to 80 percent.

(E) If small losses of test chemical were observed in SHW and pure water, report a first-order rate constant loss, $(k_p)_{loss}$. Calculate and report $(k_p)_{obs}$ for SHW and/or pure water. Calculate and report the corrected first-order rate constant for SHW and/or pure water using the relationship expressed in Equation 24:

Equation 24

$$k_p=(k_p)_{obs}-(k_p)_{loss}.$$

(F) Report the value of R calculated from Equation 4 under paragraph (c)(2)(vi)(D)(*4*) of this section.

(G) Report the values of k_{pE} and k_{DE} obtained from Equations 5 and 6, respectively under paragraph (c)(2)(vii) of this section; report the corresponding half-life calculated from Equation 22 under paragraph (d)(6)(iii)(I) of this section.

(ii) Phase 3—Indirect photoreaction with actino-meter. (A) Report the initial molar concentration of test chemical, C_o, in pure water and in SHW for each replicate and the mean value.

(B) Report the initial absorbance A°_{370} of the SNW solution.

(C) Report the initial molar concentration of PNAP of each replicate and the mean value in the actinometer. Report the concentration of phyridine used in the actinometer which was obtained from Equation 15 under paragraph (d)(2)(i) of this section.

(D) Report the time and date the photolysis experiments were started, the time and date the experiments were completed, and the elapsed photolysis time in days.

(E) For each time point t, report the separate values of the absorbance of the SHW solution, and the mean values.

(F) For each time point for the controls, report the separate values of the molar concentrations of test chemical in pure water and SHW, and the absorbance of the SHW solution, and the mean values.

(G) Tabulate and report the following date: t, $[C]^{SHW}$, $[C]^W$, A^{SNW}_{370}, [PNAP].

(H) From the data in (G), tabulate and report the following data: t, $Pn(C_o/C)_{SNW}$, $Pn(C_o/C)_W$, $[1-(A_{370}/A^\circ_{370})SNW]$, $Pn(A^\circ_{370}/A_{370})$, $Pn(C_o/C)_{PNAP}$.

(I) From the linear regression analysis of the appropriate date in step (H) in Equation 17 under paragraph (d)(2)(vi) of this section, report the slope S1 and the correlation coefficient.

(J) From the linear regression analysis of the appropriate date in step (H) in Equation 12 under paragraph (d)(1)(vii) of this section, report the slope S2 and the correlation coefficient.

(K) From the linear regression analysis of the appropriate data in step (H) in Equation 13a under paragraph (d)(1)(viii) of this section, report the slope S3 and the correlation coefficient.

(L) If loss of chemical was observed during photolysis in pure water and SHW, then report the data $Pn[C_o/C)_{corr}$, $Pn(C_o/C)_{obs}$, $Pn[C_o/C)_{loss}$ as described in paragraph (e)(2)(E) of this section. Repeat steps (H), (I), (J), (K) where applicable and report S1, S2, S3 and the corresponding correlation coefficients.

(M) Report the value of the actinometer rate constant obtained from Equation 18 under paragraph (d)(2)(ix) of this section.

(N) Report the value of k_{lo} obtained from Equation 19 under paragraph (d)(2)(x) of this section.

(O) Report the value of k_D obtained from Equation 20 under paragraph (d)(2)(xi) of this section.

(P) Report the value of $(k_{pE})_{SNW}$, obtained from Equation 14 under paragraph (d)(1)(ix) of this section, and the value of k_{pE} obtained from Equation 5a under paragraph (d)(1)(x) of this section.

(Q) Report the half-life, $t_{1/2E}$, obtained from Equation 22 under paragraph (d)(6)(iii)(I) of this section.

(f) *References.* For additional background information on this test guideline the following references should be consulted.

(1) Cooper W.J., Zika R.G. "Photochemical formation of hydrogen peroxide in surface and ground waters

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exposed to sunlight." *Science*, 220:711. (1983).

(2) Draper W.M., Crosby D.G. "The photochemical generation of hydrogen peroxide in natural waters." *Archives of Environmental Contamination and Toxicology*, 12:121. (1983).

(3) Draper, W.M. and Crosby D.G. "Solar photooxidation of pesticides in dilute hydrogen peroxide." *Journal of Agricultural and Food Chemistry*, 32:231. (1984).

(4) Draper W.M., Crosby D.G. "Hydrogen peroxide and hydroxyl radical: Intermediates in indirect photolysis reactions in water." *Journal of Agricultural and Food Chemistry*, 29:699. (1981).

(5) Dulin D., Mill T. "Development and evaluation of sunlight actinometers." *Environmental Science and Technology*, 6:815. (1982).

(6) Haag H.R., Hoigne J., Gassman E., Braun A.M. "Singlet oxygen in surface waters—Part I; Furfuryl alcohol as a trapping agent." *Chemosphere*, 13:631. (1984).

(7) Haag W.R., Hoigne J., Gassman E., Braun A.M. "Singlet oxygen in surface waters—Part II: Quantum yields of its production by some natural humic materials as a function of wavelength." *Chemosphere*, 13:641. (1984).

(8) Mill T., Winterle J.S., Fischer A., Tse D., Mabey W.R., Drossman H., Liu A., Davenport J.E. Toxic substances process data generation and protocol development. Work assignment 12, test standard development. "Section 3. Indirect photolysis." Draft final report. EPA Contract No. 68-03-2981. Environmental Research Laboratory, Office of Research and Development, EPA, Athens, GA, and Office of Toxic Substances, EPA, Washington, DC. (1984).

(9) Mill T., Mabey W.R., Bomberger D.C., Chou T.W., Hendry D.G., Smith J.H. "Laboratory protocols for evaluating the fate of organic chemicals in air and water. Chapter 3. Photolysis in water. Chapter 4. Oxidation in water." EPA 600/3-82-022. Environmental Research Laboratory, Office of Research and Development, EPA, Athens, GA. (1981).

(10) Mill T., Mabey W.R., Winterle J.S., Davenport J.E., Barich V.P., Dulin D.E., Tse D.S., Lee G. "Design and validation of screening and detailed methods for environmental processes. Apendix C. Lower-tier direct photolysis protocol." Draft final report. EPA Contract No. 68-01-6325. Office of Toxic Substances, EPA, Washington, DC. (1982).

(11) Mill T., Davenport J.E., Winterle J.S., Mabey W.R., Dossman H., Tse D., Liu A. Toxic substances process data generation and protocol development. Work assignment 12. "Appendix B. Upper-tier protocol for direct photolysis in water." Draft final report. EPA Contract No. 68-03-2981. Environmental Research Laboratory, Office of Research and Development, EPA, Athens, GA, and Office of Toxic Substances, EPA, Washington, DC. (July 1983).

(12) Winterle J.S., Mill T. Toxic substances process data generation and protocol development. Work assignment 18. "Indirect photoreaction protocol." Draft EPA special report. EPA Contract No. 68-03-2981. Environmental Research Laboratory, Office of Research and Development, EPA, Athens, GA and Office of Toxic Substances, EPA, Washington, DC. (1985).

(13) Mill T., Hendry D.G., Richardson H. "Free radical oxidants in natural waters." *Science*, 207:886. (1980).

(14) U.S. Environmental Protection Agency (USEPA), Office of Toxic Substances (OTS). "Chemical fate test guidelines. Test guideline (CG, CS-6000). Photolysis in aqueous solution." EPA-560/6-84-003. NTIS publication PB-84-233287. (1984).

(15) USEPA, OTS. "Chemical fate test guidelines. Test guildeline (CG, CS-6010). Laboratory determination of the direct photolysis reaction quantum yield in aqueous solution and sunlight photolysis." EPA-560/6-84-003. NTIS publication PB-84-233287. (1984).

(16) Wolff C.J.M., Halmans M.T.H., Van der Heijde H.B. "The formation of singlet oxygen in surface waters." *Chemosphere*, 10:59. (1981).

(17) Zepp R.G., Baughman G.L., Schlotzhauer P.F. "Comparison of photochemical behavior of various humic substances in water: I. Sunlight induced reactions of aquatic pollutants photosensitized by humic substances." *Chemosphere*, 10:109. (1981).

(18) Zepp R.G., Baughman G.L., Schlozhauer P.F. "Comparison of photochemical behavior of various humic substances in water: II. Photosensitized oxygenations." *Chemosphere*, 10:119. (1981).

(19) Zepp R.G., Cline D.M. "Rates of direct photolysis in aquatic environments." *Environmental Science and Technology*, 11:359. (1977).

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(21) Zepp R.G., Schlotzhauer P.F., Merritt S.R. "Photosensitized transformations involving electronic energy transfer in natural waters: role of humic substances." *Environmental Science and Technology*, 19:74. (1985).

2. In Part 799:

PART 799—[AMENDED]

a. The authority citation continues to read as follows:

Authority: 15 U.S.C. 2603, 2611, 2625.

b. Section 799.2475 is added, to read as follows:

§ 799.2475 2-Mercaptobenzothiazole.

(a) *Identification of test substance.* (1) 2-Mercaptobenzothiazole (MBT, CAS No. 149-30-4) shall be tested in accordance with this section.

(2) MBT of at least 98 percent purity shall be used as the test substance.

(b) *Persons required to submit study plans, conduct tests, and submit data.* All persons who manufacture (including byproduct manufacture, and import of MBT and MBT-containing articles) or process or intend to manufacture or process MBT, other than as an impurity, after October 21, 1988, to the end of the reimbursement period shall submit letters of intent to conduct testing, submit study plans, conduct tests, and submit data, or submit exemption applications as specified in this section, Subpart A of this Part, and Parts 790 and 792 of this chapter for single-phase rulemaking.

(c) *Chemical fate*—(1) *Aerobic aquatic biodegradation*—(i) *Required testing.* Aerobic aquatic biodegradation testing shall be conducted with MBT in accordance with § 796.3100 of this chapter.

(ii) *Reporting requirements.* (A) The aerobic aquatic biodegradation test shall be completed and the final report submitted to EPA within 12 months of the effective date of the final rule.

(B) An interim progress report shall be submitted to EPA 6 months after the effective date of the final rule.

(2) *Indirect photolysis-screening level test*—(i) *Required testing.* Indirect photolysis testing shall be conducted with MBT in accordance with § 795.70 of this chapter.

(ii) *Reporting requirements.* (A) The indirect photolysis test shall be completed and the final report submitted to EPA within 12 months of the effective date of the final rule.

(B) An interim progress report shall be submitted to EPA 6 months after the effective date of the final rule.

(3) *Chemical mobility*—(i) *Required testing.* Chemical mobility testing shall be conducted with MBT in accordance with § 796.2750 of this chapter.

(ii) *Reporting requirements.* (A) The chemical mobility test shall be completed and the final report submitted to EPA within 12 months of the effective date of the final rule.

(B) An interim progress report shall be submitted to EPA 6 months after the effective date of this final rule

(d) *Environmental effects*—(1) *Fish chronic toxicity*—(i) *Required testing.* (A) Chronic toxicity testing of MBT shall be conducted using rainbow trout (*Salmo gairdneri*) according to § 797.1600 of this chapter except for the provisions in paragraph (c)(6)(iv)(A) of § 797.1600.

(B) For the purpose of this section, the following provisions also apply:

(*1*) *Test substance measurement.* Prior to addition of the test substance to the dilution water, it is recommended that the test substance stock solution be analyzed to verify the concentration. After addition of the test substance, the concentration of test substance shall be measured in the test substance delivery chamber prior to beginning, and during, the test. The concentration of test substance should also be measured at the beginning of the test in each test concentration (including both replicates) and control(s), and at least once a week thereafter. Equal aliquots of test solution may be removed from each replicate chamber and pooled for analysis. If a malfunction in the delivery system is discovered, water samples shall be taken from the affected test chambers immediately and analyzed.

(*2*) *pH.* It is recommended that a pH of 7 be maintained in the test chambers.

(*3*) *Reporting.* An analysis of the stability of the stock solution for the duration of the test shall be reported.

(ii) *Reporting requirements.* (A) The fish chronic toxicity test shall be completed and the final report submitted to EPA within 12 months of the effective date of the final rule.

(B) An interim progress report shall be submitted to EPA 6 months after the effective date of the final rule.

(2) *Daphnid chronic toxicity*—(i) *Required testing.* (A) Daphnid chronic toxicity testing shall be conducted with MBT using *Daphnia magna* according to § 797.1330 of this chapter.

(B) For the purposes of this section, the following provisions also apply:

(*1*) *Test substance measurement.* Test substance concentration shall be measured in the test substance delivery chamber prior to beginning, and during, the test.

(*2*) *pH.* It is recommended that a pH of 7 be maintained in the test chambers.

(*3*) *Reporting.* An analysis of the stability of the stock solution for the duration of the test shall be reported.

(ii) *Reporting requirements.* (A) The daphnid chronic toxicity test shall be completed and the final report submitted to EPA within 12 months of the effective date of the final rule.

(B) An interim progress report shall be submitted to EPA 6 months after the effective date of the final rule.

(e) *Health effects*—(1) *Developmental toxicity testing*—(i) *Required testing.* Developmental toxicity testing shall be conducted in two mammalian species with MBT in accordance with § 798.4900 of this chapter, using the oral route of administration.

(ii) *Reporting requirements.* (A) The developmental toxicity test shall be completed and the final report submitted to EPA within 12 months of the effective date of the final rule.

(B) An interim progress report shall be submitted to EPA 6 months after the effective date of the final rule.

(2) *Reproductive toxicity*—(i) *Required testing.* Reproductive toxicity testing shall be conducted with MBT in accordance with § 798.4700 of this chapter, using the oral route of administration.

(ii) *Reporting requirements.* (A) The reproductive test shall be completed and the final report submitted to EPA within 29 months of the effective date of the final rule.

(B) Progress reports shall be submitted to EPA at 6-month intervals beginning 6 months after the effective date of the final rule until submission of the final report.

(3) *Neurotoxicity*—(i) *Required testing.* (A)(*1*) An acute and subchronic functional observation battery shall be conducted with MBT in accordance with § 798.6050 of this chapter except for the provisions in paragraphs (d)(5) and (6) of § 798.6050.

(*2*) For the purpose of this section, the following provisions also apply:

(*i*) *Duration and frequency of exposure.* For acute study, animals shall be administered MBT over a period not to exceed 24 hours. For subchronic study, animals shall be dosed daily for at least 90 days.

(*ii*) *Route of exposure.* Animals shall be exposed to MBT orally.

(B)(*1*) An acute and subchronic motor activity test shall be conducted with MBT in accordance with § 798.6200 of this chapter except for the provisions in paragraphs (d)(5) and (6) of § 798.6200.

(*2*) For the purpose of this section the following provisions also apply:

(*i*) *Duration and frequency of exposure.* For acute study, animals shall be administered over a period not to exceed 24 hours. For subchronic study, animals shall be dosed daily for at least 90 days.

(*ii*) *Route of exposure.* Animals shall be exposed to MBT orally.

(C)(*1*) A subchronic neuropathology test shall be conducted with MBT in accordance with § 798.6400 of this chapter except for the provisions in paragraphs (d)(5) and (6) of § 798.6400.

(*2*) For the purpose of this section, the following provisions also apply:

(*i*) *Duration and frequency of exposure.* Animals shall be dosed daily for at least 90 days.

(*ii*) *Route of exposure.* Animals shall be exposed to MBT orally.

(*ii*) *Reporting requirements.* (A) The functional observation battery, motor activity, and neuropathology tests shall be completed and the final reports for each test submitted to EPA within 12 months of the effective date of the final rule.

(B) A progress report shall be submitted to EPA for the functional observation battery, motor activity, and neuropathology tests, respectively, 6 months after the effective date of the final rule.

(4) *Mutagenic effects—Chromosomal aberrations*—(i) *Required testing.* (A) A dominant lethal assay shall be conducted with MBT in accordance with § 798.5450 of this chapter, using the oral of administration.

(B) A heritable translocation assay shall be conducted with MBT in accordance with the test guideline specified in § 798.5460 of this chapter if MBT produces a positive result in the dominant lethal assay conducted pursuant to paragraph (e)(4)(i)(A) of this section and if, after a public program review, EPA issues a Federal Register notice or sends a certified letter to the test sponsor specifying that the testing shall be initiated.

(ii) *Reporting requirements.* (A) Mutagenic effects—Chromosomal aberration testing of MBT shall be completed and the final report submitted to EPA as follows: Dominant lethal assay, within 12 months after the effective date of this rule; heritable translocation assay, within 24 months after notification under paragraph (e)(4)(i)(B) of this section that the testing shall be initiated.

(B) For the dominant lethal assay, an interim progress report shall be submitted to EPA 6 months after the effective date of the final rule; for the heritable translocation assay, progress reports shall be submitted to EPA at 6-month intervals beginning 6 months after the date of EPA's notification of the test sponsor that testing shall be initiated until submission of the final report.

(f) *Effective date.* (1) The effective date of this final rule is October 21, 1988.

(2) The guidelines and other test methods cited in this section are referenced here as they exist on October 21, 1988.

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[Information collection requirements have been approved by the Office of Management and Budget under Control Number 2070–0033].

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