


# BIOASSAYS OF MRI CORPORATION

# TAMPA, HILLSBOROUGH COUNTY, FLORIDA

NPDES #FL0001775

Biological Section
Bureau of Water Analysis
December 2, 1980

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## INTRODUCTION

The MRI Corporation is a tin electrowinning (recovery of metals from solution using electrolysis) plant located at 9220 Stannum St., Tampa, Hillsborough County, Florida (Fig. 1). The NPDES designated outfall (#001) is to an unnamed ditch, thence to Six Mile Creek, and ultimately to Tampa Bay (Fig. 2). Toxic substances in the discharge may include heavy metals (mercury, chromium, copper, lead, iron, aluminum, cadmium, zinc and nickel) and oil and grease. Treatment prior to discharge of 0.044 MGD wastewater includes a calcium hypochlorite rinse, acidification with sulfuric acid, rotating drum vacuum filtration, neutralization, and final dilution with non-contact cooling water (Fig. 3).

This bioassay of the MRI Corporation was requested by the Hillsborough County Environmental Protection Commission, to test the effects the effluent might have on the biota of the receiving waters. The results of these tests are presented here.

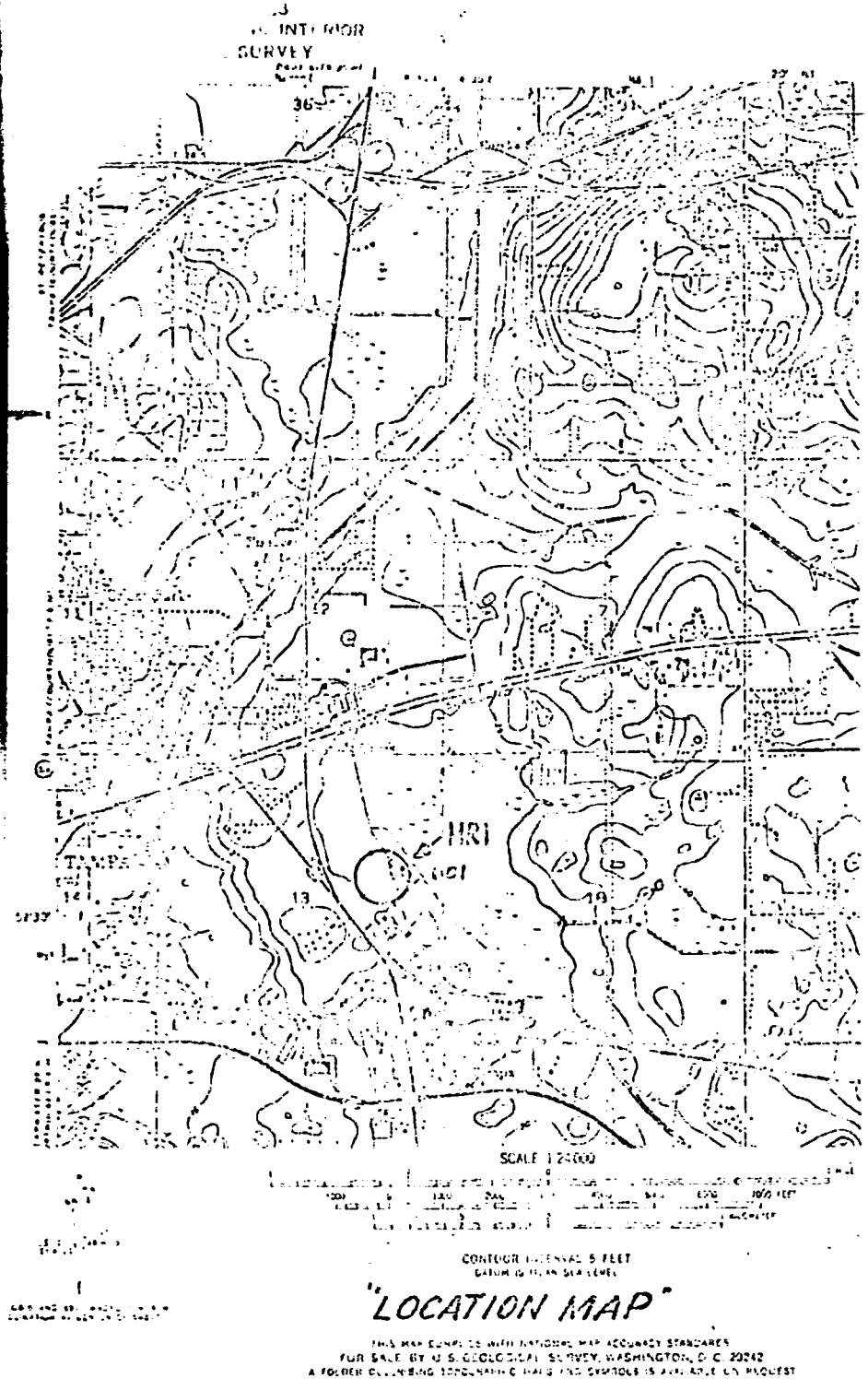



Figure 1.
MRI Corporation
9220 Stannum Street
Tampa, Florida 33619
3/12/79

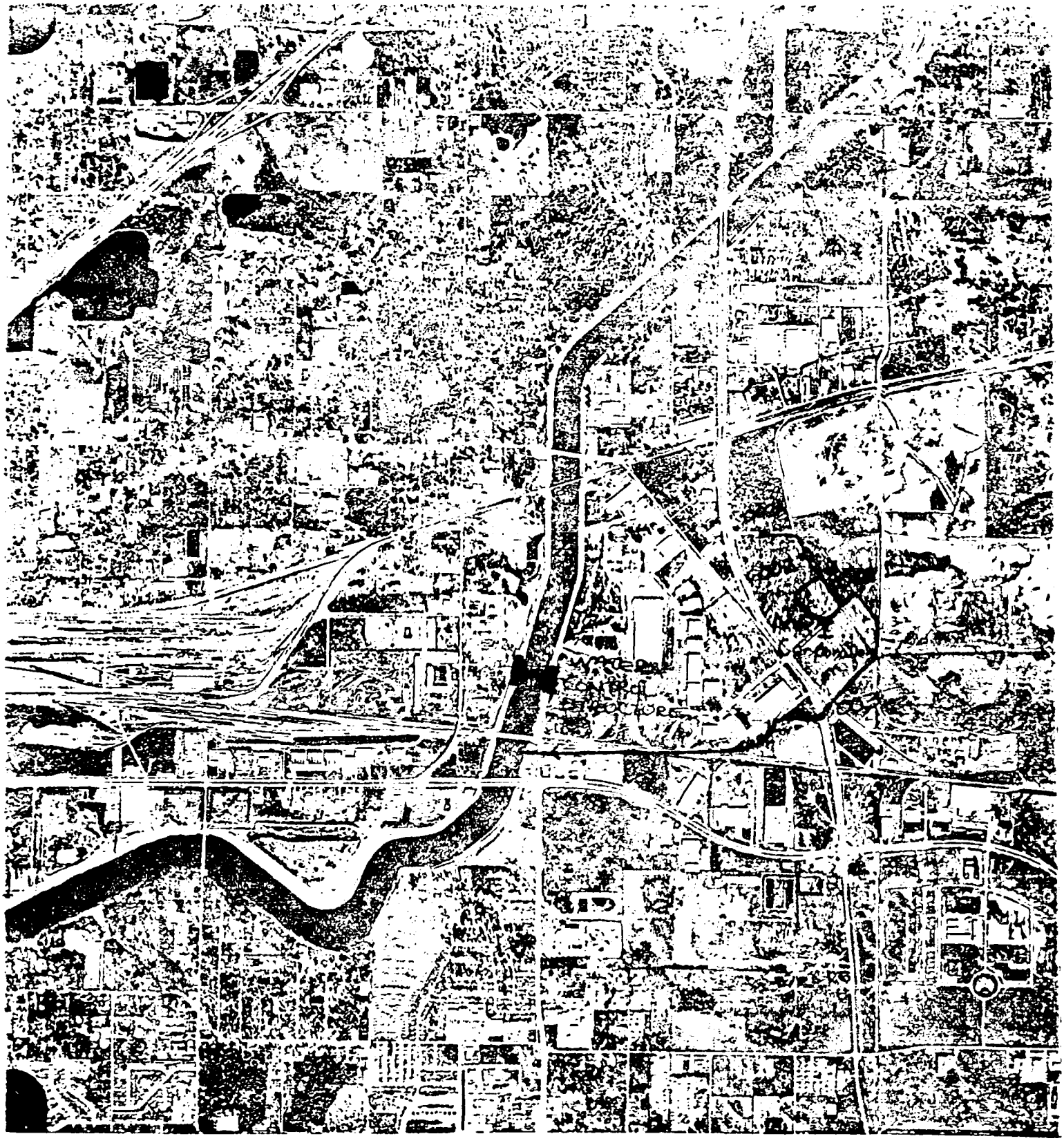


Figure 2. MRI Corporation, Route of Plant Discharge to Six Mile Creek Tampa, Florida.

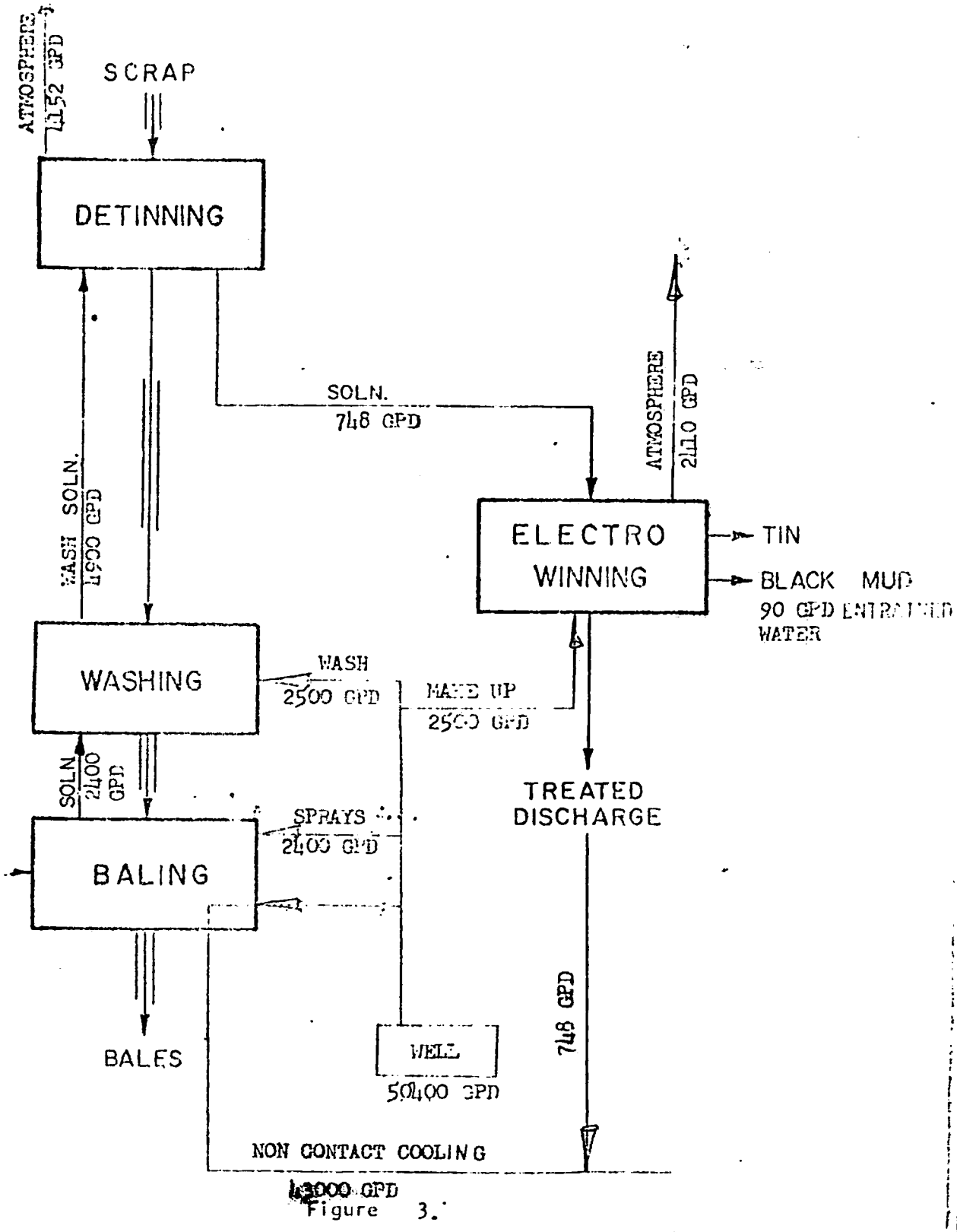



Figure 3. SCHEMATIC OF WATER FLOW

MRI Corporation
9220 Stannum Street
Tampa, Florida 33619
3/12/79

## METHODS AND MATERIALS

Test methods used to conduct these static bioassays are extensively described by Peltier (1978). Mysidopsis bahia (mysid shrimp) were selected as test organisms as recommended by Peltier (1978) and U.S. EPA (1978) for saltwater discharges. The life history and maintenance procedures used to culture them are detailed below.

### History and Maintenance of Bioassay Organisms

On 19 September 1978, approximately 300 adult mysid shrimp (Mysidopsis bahia) were obtained from the Gulf Breeze EPA Laboratory in Pensacola, Florida. The mysids have been cultured under continuous lighting in 10 and 15 gallon aquaria equipped with undergravel filtration. Seawater is obtained from the FSU Marine Laboratory located at Turkey Point, Florida (salinity range 17-31 0/oo. The mysids are fed twice daily with freshly hatched Artemia salina (brine shrimp) and kept at a constant temperature of $22^{\circ} \pm 1^{\circ}C$.

### Test Methods

Two 48-hour static bioassays were conducted from 1000 hours, 1 July 1980 to 1000 hours 3 July 1980, on a grab sample of effluent from MRI Corporation outfall #001. Grab samples were collected by Mr. Bob Carpenter of the Hillsborough County Environmental Protection Commission at 1000 hours on 30 June 1980 using standard wastewater sampling procedures. Field parameters and observations concerning the effluent, as recorded by Mr. Carpenter, are presented in Table 1. Separate samples were obtained for chemical and biological testing. Samples for metal analysis were preserved with $HNO_3$ (2 ml/liter). Samples for organic chemical analysis and bioassay tests were unpreserved. The samples were iced and transported to DER's Tallahassee Laboratory within 24 hours of collection.

Two separate bioassays using mysids were performed on the MRI Corporation effluent. One assay used saltwater collected at the FSU Marine Laboratory at Turkey Point (Salinity = 17 0/oo) for dilution of the effluent sample. In the second assay, freshly autoclaved artificial sea salts (Marinemix R) were mixed with unchlorinated well water from our well until a similar salinity was reached. Sea salts were also added to the effluent sample to minimize salinity differences between the various test concentrations (salinity was therefore maintained at 17 to 18 0/oo in all of the concentrations in both static tests).

Two replicates, each of six concentrations of effluent and a control, were used in these static tests. Immediately prior to the bioassays, a suitable number of mysids were transferred by net to a culture dish for observation and selection of individuals for the test. Mysid shrimp used in these tests were all 3/4 adult size (approximately 10 days old). Individual mysid shrimp were siphoned or pipetted via glass tube, carefully examined for injury and transferred to wide mouth quart jars, each containing 500 ml of the appropriate concentration. The concentration series selected for this study (Table 2) are those recommended by APHA (1976).

Five organisms were loaded per test chamber, giving ten per concentration. Temperature, pH, dissolved oxygen, and the number of live organisms were recorded at 0, 24, and 48 hours (Tables 3 and 4). According to U.S. EPA (1978), "Although it is generally undesirable to feed most fish and macroinvertebrates during static toxicity tests, an exception is necessary for M. bahia". Therefore, mysids were fed brine shrimp nauplii (Artemia)daily at a rate of approximately 10-20 per mysid to minimize cannibalism.

Table 1. Field Parameters and Observations

Sampling Location: Outfall # 001, end of 6" steel pipe to ditch.

Sampling Date: 30 June, 1980 10:00 A.M.

Temperature: 26°C

Conductivity: 3078 µmhos/cm

pH: 8.3

Dissolved Oxygen: 1.5 mg/l

Total Residual Chlorine: not detected

Observations: Process water normally mixed with cooling water discharge at 1.0 gpm. At time of sample process discharge at 0.5 gpm.

Table 2. Concentration Series and Volumes used for these Static Bioassays.

| Concentration | Seawater diluent | Effluent | Total Vol. |
|---|---|---|---|
| Control | 500 ml | --- | 500 ml |
| 5.6% | 472 ml | 28 ml | 500 ml |
| 10.0% | 450 ml | 50 ml | 500 ml |
| 18.0% | 410 ml | 90 ml | 500 ml |
| 32.0% | 340 ml | 160 ml | 500 ml |
| 56.0% | 220 ml | 280 ml | 500 ml |
| 100% | --- | 500 ml | 500 ml |

Table 3 . Data recorded during 48 hour, Mysidopsis bahia, static acute toxicity bioassay performed on MRI Corporation, Tampa, Hillsborough County, Florida. NPDES #FL0001775, outfall #001. 1 July to 3 July, 1980.

#1. Turkey Point Seawater

| Conc. or % | Test Container Number | Number of Live Organisms | | | | | Dissolved Oxygen (mg/l) | | | pH | | | Total Alkalinity (mg/l as $CaCO_3$) | | | | | Total Hardness (mg/l as $CaCO_3$) | | | | | Salinity (0/00) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | 0 | 24 | 48 | 72 | 96 | 0 | 24 | 48 | 0 | 24 | 48 | 0 | 24 | 48 | 72 | 96 | 0 | 24 | 48 | 72 | 96 | 0 | 24 | 48 | 72 | 96 |
| Control | A | 5 | 5 | 5 | | | 7.6 | 5.3 | 5.9 | 7.9 | 7.4 | 7.2 | | | | | | | | | | | 17 | | | | |
| Control | B | 5 | 5 | 5 | | | 7.6 | 5.9 | 6.1 | 7.9 | 7.5 | 7.2 | | | | | | | | | | | | | | | |
| 5.6% | A | 5 | 5 | 5 | | | 7.4 | 5.0 | 5.4 | 8.0 | 7.6 | 7.4 | | | | | | | | | | | | | | | |
| 5.6% | B | 5 | 5 | 5 | | | 7.3 | 5.8 | 5.4 | 8.0 | 7.6 | 7.4 | | | | | | | | | | | | | | | |
| 10.0% | A | 5 | 5 | 5 | | | 7.3 | 4.7 | 5.1 | 8.0 | 7.7 | 7.4 | | | | | | | | | | | | | | | |
| 10.0% | B | 5 | 5 | 5 | | | 7.3 | 5.2 | 5.0 | 8.0 | 7.7 | 7.4 | | | | | | | | | | | | | | | |
| 18.0% | A | 5 | 5 | 5 | | | 7.2 | 4.2 | 4.4 | 8.1 | 7.8 | 7.5 | | | | | | | | | | | | | | | |
| 18.0% | B | 5 | 5 | 4 | | | 7.2 | 4.2 | 4.7 | 8.1 | 7.9 | 7.6 | | | | | | | | | | | | | | | |
| 32.0% | A | 5 | 5 | 5 | | | 6.9 | 3.4 | 3.9 | 8.2 | 8.0 | 7.7 | | | | | | | | | | | | | | | |
| 32.0% | B | 5 | 5 | 5 | | | 6.9 | 2.7 | 3.1 | 8.2 | 8.0 | 7.8 | | | | | | | | | | | | | | | |
| 56.0% | A | 5 | 2 | 1 | | | 6.5 | 1.2 | 1.3 | 8.3 | 8.1 | 7.9 | | | | | | | | | | | | | | | |
| 56.0% | B | 5 | 5 | 5 | | | 6.4 | 2.1 | 4.4 | 8.3 | 8.2 | 7.9 | | | | | | | | | | | | | | | |
| 100% | A | 5 | 1 | 0 | | | 5.5 | 1.0 | 0.9 | 8.4 | 8.3 | 8.1 | | | | | | | | | | | 18 | | | | |
| 100% | B | 5 | 0 | 0 | | | 5.5 | 0.6 | 0.6 | 8.4 | 8.3 | 8.1 | | | | | | | | | | | | | | | |

* samples aerated to increase dissolved oxygen

Remarks:

1. Test temperature range 22 ± 2 °C.
2. 3/4 adult size (10 day old) shrimp tested
3. Free chlorine residual: none detectable.
4. Salinity of effluent adjusted to 18 0/00.

Table 4 . Data recorded during 48 hour, Mysidopsis bahia, static acute toxicity bioassay performed on MRI Corporation, Tampa., Hillsborough County, Florida. NPDES #FL0001775, outfall #001. 1 July to 3 July, 1980.
#2 Artificial Sea Water

| Conc. or % | Test Container Number | Number of Live Organisms | | | | | Dissolved Oxygen (mg/l) | | | pH | | | Total Alkalinity (mg/l as $CaCO_3$) | | | | | Total Hardness (mg/l as $CaCO_3$) | | | | | Salinity (O/oo) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | 0 | 24 | 48 | 72 | 96 | 0 | **24 | 48 | 0 | 24 | 48 | 0 | 24 | 48 | 72 | 96 | 0 | 24 | 48 | 72 | 96 | 0 | 24 | 48 | 72 | 96 |
| Control | A | 5 | 3 | 3 | | | 7.6 | 7.0 | 6.1 | 8.9 | 8.7 | 8.5 | | | | | | | | | | | 16‰ | | | | |
| Control | B | 5 | 4 | 3 | | | 7.6 | 7.2 | 6.5 | 8.9 | 8.7 | 8.5 | | | | | | | | | | | | | | | |
| 5.6% | A | 5 | 5 | 5 | | | 7.5 | 6.1 | 6.0 | 8.9 | 8.7 | 8.4 | | | | | | | | | | | | | | | |
| 5.6% | B | 5 | 2 | 2 | | | 7.5 | 6.0 | 5.4 | 8.9 | 8.7 | 8.4 | | | | | | | | | | | | | | | |
| 10.0% | A | 5 | 5 | 5 | | | 7.4 | 6.5 | 6.0 | 8.8 | 8.7 | 8.4 | | | | | | | | | | | | | | | |
| 10.0% | B | 5 | 4 | 3 | | | 7.4 | 6.5 | 5.4 | 8.8 | 8.7 | 8.4 | | | | | | | | | | | | | | | |
| 18.0% | A | 5 | 5 | 5 | | | 7.3 | 4.8 | 5.0 | 8.8 | 8.6 | 8.4 | | | | | | | | | | | | | | | |
| 18.0% | B | 5 | 5 | 5 | | | 7.3 | 5.9 | 5.3 | 8.8 | 8.6 | 8.4 | | | | | | | | | | | | | | | |
| 32.0% | A | 5 | 5 | 4 | | | 7.0 | 5.9 | 3.7 | 8.7 | 8.6 | 8.3 | | | | | | | | | | | | | | | |
| 32.0% | B | 5 | 5 | 5 | | | 7.0 | 4.2 | 3.9 | 8.7 | 8.5 | 8.3 | | | | | | | | | | | | | | | |
| 56.0% | A | 5 | 5 | 1 | | | 6.4 | 3.0 | 4.0 | 8.5 | 8.5 | 8.2 | | | | | | | | | | | 18‰ | | | | |
| 56.0% | B | 5 | 5 | 0 | | | 6.4 | 3.5 | 1.7 | 8.5 | 8.5 | 8.2 | | | | | | | | | | | | | | | |
| 100% * | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 100% * | | | | | | | | | | | | | | | | | | | | | | | | | | | |

Remarks:

1. Test temperature range 22 ± 2°C.
2. 3/4 adult size (10 day old) shrimp tested.
3. Free chlorine residual: none detectable.
4. Salinity of effluent adjusted to 18 0/00.
5. Dilution water consisted of unchlorinated well water adjusted to 18 0/00 salinity with Marinemix® sea salts.

* Due to insufficient quantities of effluent 100% concentrations were not tested.
** Samples aerated to increase dissolved oxygen.

## RESULTS AND DISCUSSION

This particular grab sample of effluent from MRI Corporation was toxic to mysid shrimp. The $LC_{50}$ was 67% at 24 hours and 60.3% at 48 hours (Table 5). (The 96 hour $LC_{50}$ would therefore be $\leq$60.3%.). Lower 48 hour $LC_{50}$ values (42.4%) were determined for the bioassay test which used artificial seawater as diluent. This test, however, was considered invalid since more than 20% of control organisms died (Table 4). Additionally, the 60% $LC_{50}$ reported here may be conservative since process water from this facility was mixed with cooling water at ½ the normal rate (0.5 GPM as opposed to 1.0 GPM) at the time of sampling (information obtained from field data sheet).

Results of chemical analyses performed on concurrent effluent grab samples from MRI Corporation are presented in Table 6. Of the parameters tested, iron and aluminum were found in quantities approaching toxic levels. These metals were found in concentrations below the levels known to be toxic to most of the species of fish tested, however, their toxicity is known to vary with pH, hardness and turbidity (U.S. EPA 1977). Cyanide, a highly toxic compound which was not tested form, may also be present in the MRI Corporation effluent. Monitoring reports submitted by the company indicate cyanide levels (reported as free cyanide amenable to chlorine) were 0.01 mg/l in June, 0.3 mg/l in July and <0.05 mg/l in August 1980 (Bob Carpenter, Personal Communication). Since levels of 0.01 to 0.05 mg/l as CN have proved fatal to many sensitive fish species (U.S. EPA 1977), it is possible that cyanide may have been responsible for the toxicity reported here.

Although it is not recommended, all test concentrations were periodically aerated to prevent mortality in the test organisms stemming from depleted dissolved oxygen. The low dissolved oxygen levels recorded at 24 and 48 hours may have contributed some additional stress to the test organisms in the 32%, 56%, and 100% concentrations (Tables 3 and 4). The cause of this dissolved oxygen depletion is attributed to the effluent since the dissolved oxygen in the lower dilutions remained relatively high. Additionally, low dissolved oxygen may have acted to increase the toxicity of any cyanide present in the effluent.

Chapter 17-3.051, Florida Administrative Code, indicates that "all waters of the State shall at all places and at all times be free from...components of discharges which...are acutely toxic." "Acute toxicity" is defined in Chapter 17-3.021(1) to mean "the presence of one or more substances...in amounts which are greater than one-third (1/3) of the amount lethal to 50% of the test organisms in 96 hours (96 hour $LC_{50}$)...". In the absence of a mixing zone, this 1/3 of the 96 hour $LC_{50}$ applies at the point of discharge. Chapter 17-.4.244(4) states that "in no event shall the maximum concentration of wastes in the mixing zone exceed the amount lethal to 50% of the test organisms in 96 hours (96 hour $LC_{50}$) for a species significant to the indigenous aquatic community..." This criterion applies at the point of discharge if there is a mixing zone. The distinction between presence or absence of a mixing zone is not relevant here, however, since the testing procedure used does not involve effluent concentration. The 1/3 of the 96 hour $LC_{50}$ criterion, is three times as stringent as the 96 hour $LC_{50}$ criterion, and therefore, in the absence of a mixing zone, the procedure used is very lenient. The procedure followed is also lenient in that testing is only done for 48 hours. The 96 hour $LC_{50}$ derived from this testing is therefore reported as "less than or equal to" the 48 hour measurement. The value found after 48 hours

can never be more restrictive than the value found after 96 hours. It must be noted that all organisms used in these tests are significant to the indigenous aquatic community since they are sensitive to toxic substances which may be expected to impact the indigenous community.

The toxicity reported here is in excess of allowable limits.

Table 5. $LC_{50}$ Data for Bioassays of MRI Corporation.

| Organism | Method of Data Analysis | 24 hour $LC_{50}$ and 95% confidence intervals | 48 hour $LC_{50}$ and 95% confidence intervals |
|---|---|---|---|
| Mysidopsis bahia using Turkey Point Seawater as diluent | Binomial | 67.1% (32-100) | 60.3% (32-100) |
| | Moving average | 66.2% (54-87) | A |
| | Probit | 66.9% (33-85) | 54.7% (0-$\infty$ ) B |
| Mysidopsis bahia using artificial sea water (unchlorinated well water adjusted for salinity with Marinemix ® sea salts as diluent). | Binomial | 79.6% (0-100) | 42.3% (0-56)* |
| | Moving average | A | 42.4% (34-53)* |
| | Probit | 172% (0-$\infty$ ) B | 31.6% (0-$\infty$ ) B* |

A. Due to the distribution of mortality, the moving average method cannot be used with this data set.

B. Due to the distribution of mortality, results calculated using the probit method are not statistically sound.

*This bioassay was considered invalid, since more than 20% of the control organisms died.

Table 6. Summary of Chemical Data for MRI Corporation on 30 June 1980.

| Parameter | Value* |
|---|---|
| Cadmium | <1 ug/l |
| Lead | <10 ug/l |
| Copper | 18 ug/l |
| Iron | 825 ug/l |
| Zinc | <25 ug/l |
| Aluminum | 1479 ug/l |
| Chromium | < 10 ug/l |
| Mercury | <0.2 ug/l |
| Nickle | <10 ug/l |
| Sodium | 71 ug/l |
| Phenols | Not Detected |
| Endosulfan II | <0.02 ug/l |
| Endosulfan Sulfate | <0.02 ug/l |

* Expressed as an average of two replicate samples.

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