MRI REPORT

ASSESS RESERVE MINING COMPANY'S SURFACE AND GROUNDWATER MONITORING AND QUALITY ASSURANCE PROGRAMS AT MILE POST 7

QUARTERLY REPORT

MRI Project No. 4558-N

December 20, 1978

For

MINNESOTA POLLUTION CONTROL AGENCY
1935 West County Road B2
Roseville, Minnesota 55113

Attention: Loren Voigt

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(2B) QUARTERLY REPORT

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environments - water
mines and minerals

PREFACE

This report presents the activities of a project sponsored by the Minnesota Pollution Control Agency, "Assess Reserve Mining Company's Surface and Groundwater Monitoring and Quality Assurance Programs at Mile Post 7." The report covers the quarter beginning July 1, 1978, and ending November 30, 1978. The data were collected from documents supplied by the Minnesota Pollution Control Agency and Reserve Mining Company. MRI personnel also made on-site visits during the weeks of October 2 and 9, 1978. Observations made during that visit are also included. The Technical Project Officer is Mr. Loren Voigt.

Ms. Janine L. Neils is project leader and was assisted by Mr. Roscoe Colingsworth and Mrs. Jean Williams. The work of organizing, analyzing and reporting is performed at the North Star Laboratories of Midwest Research Institute (MRI) by the Chemical Sciences Division.

The cooperation of personnel from MPCA and Reserve Mining Company made this project possible. MRI personnel are grateful for their efforts.

The project staff also gratefully acknowledges the assistance of Dr. Lawrence Rust, and Dr. Edwin Cook of the University of Minnesota, who acted as consultants on this report.

Chemical Sciences Division
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INTRODUCTION

The objective of this program is to provide the State of Minnesota through the Minnesota Pollution Control Agency (MPCA) with consultant services which include ongoing information on and assessment of the surface and groundwater and quality assurance program of Reserve Mining Company (Reserve) at the Mile Post 7 site. This project is stipulated by Reserve's disposal system permit (Permit No. MNCO 40509).

Midwest Research Institute personnel are charged with the assessment of: 1) the split sample program conducted between Reserve and the Minnesota Department of Health (MDH); 2) Reserve's biological monitoring program; 3) Reserve's intra-laboratory quality assurance program; and 4) Reserve's fiber monitoring program. Recommendations are also made by MRI personnel which will substantially improve the quality of these programs.

Data for these assessments were provided by the MPCA staff and Reserve Mining officials. Observations of actual sampling and analysis practices were accomplished by MRI personnel during the weeks of October 2 and 9, 1978. Biological sampling was observed and several chemical procedures were also witnessed within the laboratory.

ASSESSMENT OF THE SPLIT SAMPLE PROGRAM BETWEEN RESERVE MINING AND THE MINNESOTA DEPARTMENT OF HEALTH

Two sets of monthly data have previously been compared (February and May 1978). Data for the month of August 1978 have been made available and have been analyzed to detect differences between Reserve and MDH test results. The results for each parameter included in the split sample program are listed in Table 1. The test for differences for each parameter has been completed for each of the three months as well as for composite cumulative data for the first two months and all three months. Each test for the equality of means between Reserve and MDH has been conducted at the five percent significance level. Table 2 contains all the raw t-test data for this quarter from which the results were calculated.

Results of Pair Sample Analysis for Three Sets of Monthly Data

1. There is no significant difference between Reserve and MDH test results for each of the three sets of monthly data or for the two- and three-month composite test results for the parameters listed in Table 3.

2. Insufficient data exists to use the paired-difference test for eight parameters. In most cases, the parameter level is below the lower detection limit for Reserve, MDH, or both. In these cases, the reported value has been indicated as a "less than" value (e.g.: <0.2). Even though a formal test for differences cannot be made, each pair of values can be compared for consistency. For example, if Reserve reports a value of 0.05 and MDH reports a value of <0.2, these results are consistent with one another. Conversely, if Reserve reports a value of 2.0 and MDH reports <1.0, the results are inconsistent. A summary of the eight parameters is presented in Table 4. The number of consistent and inconsistent pairs is also presented.

3. Six parameters exhibit no significant difference between Reserve and MDH when all three months of data are combined. However, significant

Table 1. Comparative Data Between Reserve Mining (RMC) and the Minnesota Department of Health (MDH) from August 1978 (Surface Waters)

	Station 103		Station 106		Station 111		Station 112	
	RMC	MDH	RMC	MDH	RMC	MDH	RMC	MDH
Calcium	16	15	15	14	12	13	11	11
Magnesium	4.3	3.6	4.6	4.1	4.0	3.4	3.8	3.2
Sodium	1.5	1.6	2.0	1.8	1.8	1.6	1.8	1.6
Potassium	.25	<.5	.48	<.5	.22	<.5	.28	<.5
Sulfate	2.6	6.9	3.3	6.7	3.3	7.0	2.4	7.0
Chloride	<1	1.7	<1	1.3	<1	1.3	<1	1.2
Nitrate-N	<.10	.02	<.10	<.01	<.10	.04	.11	.05
Silica	9.5	11	7.7	9.0	9.1	7.5	8.9	11
Organic-N	.92	1.1	.93	.98	.93	1.0	.91	.99
Sol. O-PO_4	.005	.011	.005	.011	.003	.008	.006	.008
Total PO_4	.031	.036	.027	.028	.025	.023	.025	.024
Iron µg/l	1390	1700	1000	1300	830	1200	1030	1300
Manganese µg/l	190	240	8	20	20	37	17	30
Copper µg/l	1.2	1.4	1.9	2.5	.8	1.2	1.7	1.4
Zinc µg/l	1.8	2.5	1.7	2.4	1.2	2.3	1.4	3.1
Nickel µg/l	3	<2	3	<2.0	3	<2	4	<2
Cadmium µg/l	<.2	.023	<.2	.025	<.2	.020	<.2	.015
Lead µg/l	<2	.21	<2	.19	<2	.18	<2	.15
Cobalt µg/l	<1	.8	<1	<.5	<1	<.5	<1	.5
Chromium µg/l	1	.6	<1	.8	<1	.6	<1	.6
Silver	<1	<.02	<1	<.02	<1	<.02	<1	<.02
Arsenic µg/l	<5	.8	<5	1.3	<5	.9	<5	.7
Selenium µg/l	<5	<1	<5	<1	<5	<1	<5	<1
Barium µg/l	<10	<15	<10	<15	<10	<15	<10	<15
Fluoride	<.5	.13	<.5	.13	<.5	.13	<.5	.13
TSS	5.1	7.6	1.6	1.2	1.0	2.4	.7	1.2
COD	58	61	55	59	59	63	58	61
Color	120	180	135	160	130	160	140	160
pH	7.1	7.0	8.0	7.8	7.7	7.2	7.6	7.3
TDS	68	100	67	93	54	100	52	100
Turbidity	7.5	3.0	2.6	2.5	1.6	2.0	1.7	1.5
Ammonia-N	.121	<.2	.076	<.2	.157	<.2	.082	<.2
Spec. Cond.	104	100	103	100	83	90	80	90
Cyanide	<.01	<.001	<.01	<.001	<.01	<.001	<.01	<.001
Mercury	<.2	<.1	<.2	<.1	<.2	<.1	<.2	<.1

Table 1. Comparative Data Between Reserve Mining (RMC) and the Minnesota Department of Health (MDH) from August 1978 (Ground Waters) continued

	60M		11AM		11M		3M		9M		6M	
	RMC	MDH	RMC	MDH	RMC	MDH	RMC	MDH	RMC	MDH	RMC	MDH
Calcium	39.8	38	32	21	21	30	43	34	36	36	72	64
Magnesium	13.8	13	8.6	12	12.8	7.8	0.68	<2	8.6	8.0	20	20
Sodium	12.8	11	4.6	4.3	4.4	4.4	18	18	4.2	3.9	13	13
Potassium	0.45	<0.5	0.3	<0.5	<0.05	<0.5	0.84	0.58	0.28	<0.5	0.45	<0.5
Sulfate	13.7	11	9.6	6.3	8.2	7.4	8.7	7.4	9.2	7.8	14	10
Chloride	1.4	1.8	4.0	0.73	<1.0	0.89	<1.0	0.94	<1	0.71	1.6	1.8
Nitrate-N	<0.1	0.05	0.17	0.12	<0.10	0.20	0.11	0.10	0.19	0.18	0.70	0.70
Silica	13.7	14	20	13	14	18	16	18	18	17	219	21
Organic-N												
Sol. O-PO_4	0.003	0.002	0.003	<0.001	0.002	<0.001	0.001	<0.001	0.003	<0.001	0.003	<0.001
Total PO_4												
Iron µg/l	4	62	4	<20	4	<20	4	<20	4	<20	4	22
Manganese µg/l	111	120	1	5	<1	14	<1	<3	3	4	3	8
Copper µg/l	4.5	4.5	9.6	8.0	1.5	2.6	2.2	2.6	0.9	1.0	6.4	9.7
Zinc µg/l	3.1	5.2	2.4	13	1.8	11	2.2	0.44	2.2	3.3	9.9	8.0
Nickel µg/l	3.0	2.0	<2	<2.0	<2	3.9	<2	<2.0	<2	<2.0	<2.0	<2.0
Cadmium µg/l	<0.2	0.46	<0.2	0.23	<0.2	0.22	<0.2	<0.01	<0.2	0.053	0.4	0.32
Lead µg/l	<0.2	0.25	2	0.23	2	0.43	<2	0.28	<2	0.13	<2	0.19
Cobalt µg/l	<1	1.1	<1	0.7	<1	<0.5	<1	0.7	<1	<0.5	<1	0.9
Chromium µg/l	<1	<0.5	1	1.0	12	1.3	18	3.9	2	0.5	2	1.6
Silver µg/l												
Arsenic µg/l												
Selenium µg/l												
Barium µg/l	<10	<15	<10	<15	<10	<15	<10	39	<10	<15	<10	20
Fluoride	<0.5	0.26	<0.5	0.13	<0.5	<0.1	<0.5	<0.1	<0.5	<0.1	<0.5	0.18
TSS												
COD	6.5	<5	4.5	<5	1.2	8	3.3	<5	2.8	6	—	8
Color	<5	<5	5	<5	5	<5	<5	<5	<5	<5	<5	<5
pH	7.97	7.8	7.72	8.0	8.15	7.8	11.4	11	7.95	8.2	7.68	7.9
TDS	227	190	156	130	146	140	390	89	169	150	361	86
Turbidity												
Ammonia-N	0.063	<0.2	<0.01	<0.2	<0.01	<0.2	0.054	<0.2	<0.01	<0.2	0.004	<0.2
Spec. Cond.	350	340	240	210	225	220	600	200	260	240	555	490

TABLE 2. T-TEST DATA FROM THE SPLIT SAMPLE PROGRAM
Cumulative and August Comparisons

PARAMETER	AUGUST N	AUGUST $\bar{D}$	AUGUST S	AUGUST T	AUGUST RMC =, ≠ MDH at 5% Significant Level	COMBINED N	COMBINED $\bar{D}$	COMBINED S	COMBINED T	COMBINED RMC =, ≠ MDH at 5% Significant Level
CALCIUM	10	2.18	5.828	1.183	R=M	29	0.317	4.408	0.387	R=M
MAGNESIUM	9	0.6	2.05	0.8780	R=M	23	0.5443	1.8304	1.4261	R=M
SODIUM	10	0.29	0.5486	1.5858	R=M	29	0.643	1.8849	1.8371	R=M
POTASSIUM	1	0.26	---	---	---	14	-0.3179	1.3077	-0.9096	R=M
SULFATE	10	-0.25	3.3788	-0.2340	R=M	29	-0.4552	2.7204	-0.9011	R=M
CHLORIDE	3	0.89	2.0636	0.7470	R=M	14	-1.795	6.8012	-0.9875	R=M
NITRATE-N	5	0.026	0.0270	2.1518	R=M	16	0.01	0.0618	0.6478	R=M
SILICA	10	-0.07	3.0141	-0.0734	R=M	29	-0.3207	2.1564	-0.8009	R=M
SOL. O-PO_4	5	-0.0032	0.00295	-2.4259	R=M	23	-0.0013	0.00427	-1.4654	R=M
IRON	6	-0.221	0.1460	-3.7085	R≠M	14	-0.0774	0.1671	-1.7328	R=M
MANGANESE	8	-13.875	15.5144	-2.5295	R≠M	23	-5.9565	10.6450	-2.6835	R≠M
COPPER	10	-0.42	1.2345	-1.0759	R=M	27	-0.2593	0.9262	-1.4547	R=M
ZINC	10	-2.354	4.2031	-1.7711	R=M	29	-1.3741	2.6634	-2.7783	R≠M
CADMIUM	1	0.12	---	---	---	7	0.0421	0.2292	0.4860	R=M
CHROMIUM	6	4.5167	6.2204	1.7786	R=M	8	3.3	5.7196	1.6319	R=M
BARIUM	0	---	---	---	---	5	-3.4	10.0648	-0.7554	R=M
FLUORIDE	0	---	---	---	---	7	0.02857	0.03487	2.1692	R=M
TDS	10	51.2	128.484	1.2601	R=M	29	23.745	79.476	1.6089	R=M
COD	6	-4.0	1.4478	-6.7677	R≠M	17	-2.0882	3.7490	-2.2966	R≠M
COLOR	4	33.75	17.9699	-3.7563	R≠M	11	-17.7273	18.2159	-3.2277	R≠M
pH	10	0.127	0.2847	1.4107	R=M	29	0.3062	0.4247	3.8822	R≠M
SPEC. COND.	10	52	124.179	1.3242	R=M	29	23.138	76.3328	1.6324	R=M
TOT. PO_4	4	-0.00025	0.003096	-0.4845	R=M	11	-0.019	0.03369	-1.8705	R=M
TSS	4	-1.0	1.2410	-1.6116	R=M	11	-0.4091	1.5389	-0.8817	R=M
TURBIDITY	4	1.1	2.2818	0.9641	R=M	11	0.3727	1.9794	0.6245	R=M
ORGANIC N	4	-0.095	0.05802	-3.2746	R≠M	8	-0.071625	0.07287	-2.7801	R≠M
LEAD	2	1.67	0.1414	16.7	R≠M	2	1.67	0.1414	16.7	R≠M

Table 3. Parameters for Which No Significant Differences Exist in Monthly or Combined Data

Parameter	Number of Paired Samples Available			
	February	May	August	Total
Potassium	3	10	1	14
Sulfate	9	10	10	29
Chloride	7	4	3	14
Nitrate-N	3	8	5	16
Sol. O-PO_4	8	10	5	23
Copper	7	10	10	27
Cadmium	1	5	1	7
Chromium	2	0	6	8
Barium	3	2	0	5
Fluoride	4	3	0	7
TSS	3	4	4	11
TDS	9	10	10	29
Total Phosphorus	3	4	4	11
Turbidity	3	4	4	11

Table 4. Parameters for Which Data is Unsuitable to Test for Difference in Means

Parameter	Number of Paired Values that Are:	
	Consistent	Inconsistent
Ammonia-N	29	0
Nickel	21	8
Cobalt	26	3
Mercury	7	0
Silver	11	0
Arsenic	11	0
Selenium	11	0
Cyanide	7	0

differences do exist for one or more of the monthly data sets. These six parameters are presented in Table 5 along with a description of the individual monthly differences and February/May composite differences.

In this table, the following notation has been used:

R = M: No significant difference between Reserve and MDH.
R > M: Reserve values consistently larger than MDH values.
R < M: Reserve values consistently smaller than MDH values.

4. Seven parameters exhibit significant differences between Reserve and MDH when all three months of data are combined. These parameters are presented in Table 6 in the same format as Table 5.

Table 5. Parameters for Which Composite of Three Months of Data Indicate No Difference But with One or More Sets of Monthly Data Indicating Significant Differences at the 5% Significance Level

Parameter	Type of Difference For:				
	February	May	February and May	August	February, May and August
Calcium	R > M	R < M	R = M	R = M	R = M
Magnesium	R > M	R = M	R = M	R = M	R = M
Sodium	R > M	R = M	R = M	R = M	R = M
Silica	R > M	R < M	R = M	R = M	R = M
Iron	R = M	R = M	R = M	R < M	R = M
Spec. Cond.	R > M	R = M	R = M	R = M	R = M

Table 6. Parameters for Which Composite of Three Months of Data Indicate Significant Differences Between Reserve and MDH

Parameter	Type of Difference For:				
	February	May	February and May	August	February, May and August
Magnanese	R = M	R < M	R < M	R < M	R < M
Zinc	R < M	R = M	R < M	R = M	R < M
Lead*	a	a	a	R > M	R > M
COD	R = M	R = M	R = M	R < M	R < M
Color	R = M	R = M	R = M	R < M	R < M
pH	R > M	R > M	R > M	R = M	R > M
Organic-N	a	R = M	R = M	R < M	R < M

*Based on only two paired difference values.

[a]No paired difference values available.

BIOLOGICAL MONITORING

Reserve's biological monitoring program as modified since MRI's Quarterly Report (July 18, 1978) has been reviewed. Reserve's sampling station locations are satisfactory and the sampling and analytical procedures are carefully conducted and they generally follow standard methodologies. Where Reserve's procedures vary with these methodologies, their personnel have adequately demonstrated that the variances have no observable affect on the data, except for the spectrophotometer used in the pigment analyses. Two of the macroinvertebrate samples collected this fall were analyzed in our laboratory. Both the counts and identification are in general the same.

Field Sampling

Reserve has taken steps to insure that the sampling sites at each station are as uniform as possible with regard to substrate. Stations 103 and 104 (redesignated now as 113) have both been relocated; they are now in areas of rubble-gravel-fine sand substrate. Reserve has added two control stations: Station 112 on the Beaver River upstream from its confluence with Diversion 2 (of Big Thirtynine Creek) and Station 114 on Big Thirtynine Creek upstream from its confluence with Diversion 1 (the low-flow diversion of Little Thirtynine Creek). These latter two stations appear to be well-located with regard to the construction activities and the other stations; they have not yet been visited by MRI personnel.

An evaluation of the number of Surber net samples to be collected at each station to obtain a reasonable estimate of the macroinvertebrate populations was conducted by Reserve (data included in J. Oxberry's memo of 31 August 1978 to A.H. Samuel). The analysis adequately indicates that collecting more than three samples per station does not markedly improve the estimate of the population mean.

Analysis--Macroinvertebrates

Both a qualitative and quantitative sample were sent to MRI for verification of Reserve's macroinvertebrate taxonomic work. The samples came from Stations 105 and 109, respectively. There is generally a good agreement between Reserve's and MRI's macroinvertebrate identifications. Individuals of uncertain identity were verified by Dr. Edwin F. Cook of the University of Minnesota.

At Station 109, the greatest difference in macroinvertebrate counts was in the Oligochaeta (see Table 7). This difference may have arisen by enumerating parts of individuals which had probably been broken as they were handled.

There were also some differences in the identifications of some of the Plecoptera and Ephemeroptera. Most of the individuals in question were such early instars that positive identification was extremely difficult.

The Isotomurus (Collembola) specimen in the sample from Station 105 was identified by Dr. Cook as Heteromurus (a terrestrial species). This specimen can be identified as Isotomurus if a key to only the semi-aquatic Collembola is used. However, a specialized key for all Collembola incorporates taxonomic characters (the most notable being the apparent fusion of the last abdominal segments) that place the specimen in the family Entomobryidae and the genus Heteromurus. A similar difference arises with Reserve's identification of the Limnephilid Drusinus in the qualitative sample. Many taxonomists now feel that the subfamily Drusinae is not found in North America and that "Drusinus" when found here is actually Pseudostenophylax. It would probably be of benefit to Reserve's taxonomists if their collection of keys could be expanded to include some more recent, specialized works. Some suggested keys are: H.G. Scott's *Pictorial Keys to the Nearctic Genera of Collembola* (1961); S.W. Hitchcock's *The Plecoptera or Stoneflies of Connecticut* (1974); Edmunds, Jensen, and Berner's *Mayflies of North and Central America* (1976); and G. Wiggins' *Larvae of North American Caddisfly Genera* (1977).

Table 7. Comparison of Identifications of Macroinvertebrates Collected by Reserve on October 2, 1978, at Station 109--Surber 1

Organism	RMC Number	RMC Genera Observed	Organism	MRI Number	MRI Genera Observed
Diptera			Diptera		
Chironomidae larvae	122	Not identified	Chironomidae larvae	121	Not identified
Chironomidae adults (?)	1	Not identified	Chironomidae adults	1	Not identified
Empididae	2	Hemerodromia	Empididae	2	Hemerodromia
Trichoptera			Trichoptera		
Hydropsychidae	3	Hydropsyche	Hydropsychidae	3	Hydropsyche
Rhyacophilidae larvae	16	Glossosoma	Rhyacophilidae larvae	16	Glossosoma
Rhyacophilidae pupae	7	Glossosoma	Rhyacophilidae pupae	8	Glossosoma
Ephemeroptera			Ephemeroptera		
Heptageniidae	8	Iron	Heptageniidae	8	Iron
	5	Rithrogena		5	Rithrogena
Baetidae	9	Paraleptophlebia	Baetidae	8	Paraleptophlebia
	2	Pseudocloeon		2	Pseudocloeon
	4	Baetis		5	Baetis
	14	Probably Baetis and/or Pseudocloeon		16	Probably Baetis*
Plecoptera			Plecoptera		
Nemouridae	41	Paracapnia (?)	Nemouridae	50	Paracapnia
	1	Brachyptera*		4	Probably Paracapnia*
	8	Not identified*			
Early instars	7	Not identified			
Hemiptera			Hemiptera		
Adults	6	Not identified	Aphidoidea	6	Aphids (terrestrial)
Hirudinea			Hirudinea		
Erpobdellidae	2	Dina	Erpobdellidae	2	Dina

*Early instars and/or missing parts.

*Early instars and/or missing parts.

Table 7. Comparison of Identifications of Macroinvertebrates Collected by Reserve on October 2, 1978, at Station 109--Surber 1 (Continued)

Organism	RMC Number	Genera Observed
Coleoptera		
Elmidae	13	Optioservus
Oligochaeta	27	Not identified**
Hydracarina	1	Not identified
Collembola		
Isotomidae	1	Isotomurus
Total Individuals	300	

**Broken pieces; estimated 20 individuals in sample.

Organism	MRI Number	Genera Observed
Coleoptera		
Elmidae	13	Optioservus
Oligochaeta	41	Not identified**
Hydracarina	1	Not identified
Collembola		
Entomobryidae	1	Heteromurus (terrestrial)
Total Individuals	313	

**Broken pieces; estimated 22 individuals in sample.

The qualitative sample from Station 105 contained a very large number of macroinvertebrates (see Table 8). In earlier conversations with Reserve personnel, MRI indicated that it was probably not necessary to get an exact count of the Chironomidae in the qualitative samples because quantitative samples are also collected. The final decision about this, however, is left to MPCA. As with the previous sample, it appears that most of the differences in macroinvertebrate identifications are attributable to the fact that there were many early instar Plecoptera and Ephemeroptera nymphs. The differences in counts are most likely due to MRI's finding more very small specimens as the Chironomidae were examined. Very careful examination of the "Chironomidae" for other taxa is therefore needed if the midges are not counted in the qualitative samples.

Reserve has not yet established a verification system for the macroinvertebrates. This should be done. The consultant selected for the verification program also could be of assistance in recommending the additional keys that would be helpful in recognizing northeastern Minnesota macroinvertebrates.

Analysis-Periphyton

The effects of several deviations from the standard analytical methodology for periphyton pigment analysis were analyzed by Reserve (see J. Oxberry's memo of 31 August 1978 to A.H. Samuel). Their results adequately demonstrate the acceptability of their previous practices of:

a. maintaining periphyton samples in iced coolers for the time period which usually elapses between field collection, and filtration and freezing in the laboratory; and

b. using a glass stirring rod to mix the samples during pigment extraction.

They also showed that the narrow-band instrument (Bausch and Lomb Spectronic 710) resulted in significantly higher pigment concentrations than the Spectronic 100 they had been using. Considering the change in equipment, Reserve personnel should acquire and analyze chlorophyll *a* spiked samples which are available through the EPA.

Table 8. Comparison of Identifications of Macroinvertebrates Collected by Reserve on October 4, 1978, at Station 105--Qualitative Sample

Organism	RMC Number	Genera Observed
Diptera		
Chironomidae	Many	Not identified
Ceratopogonidae	1	Probezzia, Bezzia
Simuliidae	1	Not identified
Tabanidae	1	Chrysops
Tipulidae	6	Hexatoma
Empididae	9	Hemerodromia
Rhagionidae	1	Atherix
Adult diptera	1	Not identified
Trichoptera		
Hydropsychidae	115	Hydropsyche
	27	Cheumatopsyche
	1	Not identified*
Leptoceridae	1	Mystacides**
	1	Oecetis
Hydroptilidae	14	Ochrotrichia
	13	Oxyethira
Helicopsychidae	3	Helicopsyche
Lepidostomatidae	3	Lepidostoma
Limnephilidae	1	Platycentropus (young)
	2	Drusinus
	8	Not identified*
	2	Not identified***
Ephemeroptera		
Heptageniidae	52	Stenonema
	27	Some probably Stenonema****
	1	Iron

*Early instars.
**Has sand grain case.
***Have confusing characters.
****Missing parts.

Organism	MRI Number	Genera Observed
Diptera		
Chironomidae	1336	Not identified
Ceratopogonidae	1	Bezzia, Probezzia, or Palpomyia
Simuliidae larvae	1	Simulium
Simuliidae adults	1	Simulium
Tabanidae	1	Chrysops
Tipulidae	6	Hexatoma
Empididae	11	Hemerodromia
Rhagionidae	1	Atherix
Trichoptera		
Hydropsychidae	119	Hydropsyche
	29	Cheumatopsyche
	2	Not identified*
Leptoceridae	1	Oecetis
Hydroptilidae	15	Ochrotrichia
	14	Oxyethira
Helicopsychidae	3	Helicopsyche
Lepidostomatidae	3	Lepidostoma
Philopotamidae	1	Chimarra
Limnephilidae	1	Platycentropus
	4	Pycnopsyche
	9	Probably Pycnopsyche*
Polycentropidae	1	Psychomyia
Ephemeroptera		
Heptageniidae	53	Stenonema
	32	Probably Stenonema**
	1	Iron

*Early instars.
**Missing parts.

Table 8. Comparison of Identifications of Macroinvertebrates Collected by Reserve on October 4, 1978, at Station 105--Qualitative Sample (Continued)

Organism	RMC Number	RMC Genera Observed	Organism	MRI Number	MRI Genera Observed
Ephemeroptera (cont'd)			Ephemeroptera (cont'd)		
Baetidae	16	Ephemerella	Baetidae	18	Ephemerella
	10	Caenis		9	Caenis
	5	Pseudocloeon		5	Pseudocloeon
	5	Leptophlebia		14	Leptophlebia
	32	Paraleptophlebia		36	Probably Leptophlebia**
	11	Probably Baetis or Pseudocloeon*		41	Probably Paraleptophlebia**
				8	Baetinae*
	43	Mostly Leptophlebiinae****			
	5	Baetis or Neocloeon			
Plecoptera			Plecoptera		
Perlidae	29	Acroneuria	Perlidae	29	Acroneuria
	6	Paragnetina		5	Paragnetina
Pteronarcidae	4	Pteronarcys	Pteronarcidae	4	Pteronarcys
Perlodidae	4	Isoperla (?)	Perlodidae	10	Isoperla
	5	Isoperla*			
Perlodidae or Chloroperlidae	2	Not identified			
Nemouridae	3	Taeniopteryx	Nemouridae	3	Taeniopteryx
	2	Brachyptera		1	Brachyptera
	48	Paracapnia and/or Nemoura*		58	Capniinae*,**
Unknown Plecoptera	5	Not identified			
Odonata			Odonata		
Aeschnidae	3	Boyeria Vinosa	Aeschnidae	3	Boyeria
Gomphidae	2	Ophiogomphus	Gomphidae	2	Ophiogomphus
Hemiptera			Hemiptera		
Corixidae	6	Sigara (3 males, 3 females)	Corixidae	6	Sigara (3 males, 3 females)
				1	Hesperocorixa (female)
	3	Callicorixa or Hesperocorixa (1 male, 2 females)		1	Callicorixa (male)
				1	Sigara or Callicorixa (female)

*Early instars. ***Has confusing characters.
**Has sand grain case. ****Missing parts.

*Early instars.
**Missing parts.

Table 8. Comparison of Identifications of Macroinvertebrates Collected by Reserve on October 4, 1978, at Station 105--Qualitative Sample (Continued)

Organism	RMC Number	Genera Observed	Organism	MRI Number	Genera Observed
Pelecypoda			Pelecypoda		
Sphaeriidae	2	Sphaerium	Sphaeriidae	3	Sphaerium (empty shells)
	3	Sphaerium (empty shells)			
Gastropoda			Gastropoda		
Planorbidae	1	Helisoma	Planorbidae	2	Helisoma
	1	Gyraulus			
Physidae	2	Physa (1 is empty shell)	Physidae	2	Physa (1 is empty shell)
Ancylidae	4	Ferrisia	Ancylidae	5	Ferrisia
Lymnaeidae	1	Lymnaea or Bulimnea	Amnicolidae	1	Amnicola
Coleoptera			Coleoptera		
Elmidae larvae	196	Optioservus	Elmidae larvae	214	Optioservus
Elmidae adults	15	Optioservus	Elmidae adults	15	Optioservus
	5	Dubiraphia or Vittata		5	Dubiraphia
Oligochaeta	--	Broken pieces; not identified	Oligochaeta	41	Broken pieces, not identified
Hydracarina	141	Not identified	Hydracarina	139	Not identified
Ostracoda	6	Not identified	Ostracoda	5	Not identified
Nematoda	5	Not identified	Nematoda	5	Not identified
Amphipoda			Amphipoda		
Talitridae	1	Hyallela	Talitridae	1	Hyallela
Collembola			Collembola		
Isotomidae	5	Isotomurus	Entomobryidae	5	Probably Heteromurus
Hirudinea	1	Not identified (young)			

Table 8. Comparison of Identifications of Macroinvertebrates Collected by Reserve on October 4, 1978, at Station 105--Qualitative Sample (Continued)

Organism	RMC Number	RMC Genera Observed	Organism	MRI Number	MRI Genera Observed
Fish			Fish		
Cyprinidae	2	Not identified (minnows)	Cyprinidae	1	Not identified (minnow)
			Percidae	1	Not identified (darter)
Eggs			Eggs		
Single egg cases	--	Not identified	Single egg cases	14	Not identified (possibly Hemiptera)
Egg clusters	--	Not identified	Egg clusters	2	Not identified
Total Individuals (excluding Chironomidae)	931		Total Individuals (excluding Chironomidae)	1026	

Comments and Recommendations

1. The monitoring program contains an adequate set of control stations, and all biological stations are reasonably located to minimize differences due to the type of substrate. We recommend that next year our biologist visit those sites which were not visited in 1978.

2. Three samples appear to be an adequate number of samples from each station for monitoring macroinvertebrate populations.

3. It does not appear to be necessary that Reserve count the Chironomidae in qualitative samples. However, the individuals should be carefully examined so that very early instar, non-Chironomid larvae are not missed.

4. A program to confirm Reserve's macroinvertebrate identifications should be established.

5. It does not appear necessary for Reserve to filter and freeze periphyton pigment samples in the field, nor to use a tissue grinder during pigment extraction in the laboratory.

6. The narrow-band Bausch and Lomb Spectronic 710 is recommended for measuring pigment concentrations.

7. Reserve should consider, with the help of a specialist (possibly the taxonomic confirmation consultant), expanding and updating its collection of taxonomic keys to include other keys that better apply to northeastern Minnesota.

8. Reserve personnel should consider acquiring EPA check samples for chlorophyll a analyses.

INTRA-LAB QUALITY ASSURANCE

On October 12 and 13, 1978, Ms. Janine Neils of MRI made a site visit to the Reserve Mining Company's analytical laboratory. The week after sampling was chosen so that she would be able to observe the actual performance of analyses that were scheduled for that time period. No sampling was observed due to the similarity between the September and June sampling conditions when observation was accomplished. During the winter quarter when sampling conditions differ (e.g., sampling through ice), sampling techniques will again be observed.

Compliance to Time Limits Upon Initiation of Analyses

It was suggested in the July report that Reserve laboratory order the priority of analyses along EPA guidelines. This would assure that the possibility of changes occuring within the sample be eliminated. According to observation, this has been accomplished. A chart has been compiled prioritizing the various analyses. MRI personnel are in agreement with the prioritization of analyses and time frame contained in this schedule

Methods for Analysis

Procedures actually observed while MRI personnel were in the Reserve Mining laboratory in October were: oil and grease, chemical oxygen demand, ammonia and total Kjeldahl nitrogen, chloride and all the metal analyses. All aspects of these analyses were found to be acceptable in relation to recognized federally approved techniques.

Sulfuric acid had been deleted from the digestion step for the metals analyses per MRI's recommendation. Also a more vigorous digestion step had been adopted in the analysis for mercury.

At the time of the site visit all dissolved solids data were still being reported as a calculation derived from specific conductance. It was agreed that this would prove the preferred method for surface waters due to their low concentration of dissolved solids and the high probability of error with the approved method. It was suggested that groundwaters, which had higher concentration of dissolved solids, hence lower error percentage, be run by the gravimetric method. Reserve personnel stated that although the calculated results for groundwaters were reported, both methods of analysis had been performed and would be compared. This data was not available at the time of the site visit. MRI personnel would be interested in viewing this data for possible changes in their recommendation.

Reserve Mining and MPCA officials discussed possible methods for nitrate analyses. Although MRI's position on the federally approved methods has not changed, there is agreement between Reserve and the MPCA on adopting the UV spectrophotometric technique for nitrate analyses.

Biochemical oxygen demand has been dropped as a required analysis. Data for this parameter no longer is listed on the report sheets issued by Reserve Mining and therefore will not be considered in any future reports.

Comparison of the methods used by the Minnesota Department of Health and Reserve Mining along with the minimum detectable limits for each parameter is being prepared. At the time of this report all the data had not been received. It is anticipated that this comparison will be included in next quarter's report.

Laboratory Quality Assurance

At the time of the site visit the agreed upon quality assurance program had not yet been initiated. The program was still in the planning stages. The duplication of one sample in each batch of every parameter had been begun along with the already existing duplicate sample collection and analysis. No

analyses of spiked samples or graphic representation of the duplicate data existed at that time.

EPA considers a working quality assurance program essential to the proper operation of a laboratory. MRI concurs with this belief and recommends that the Reserve Mining laboratory initiate a program similar to that outlined in the first quarterly report issued in July. This program should be initiated as soon as possible.

Recommendations for Reserve's Intra-Laboratory Quality Assurance Program

Generally the recommendations in the July report were either acted upon or an agreement was arrived at by Reserve Mining and MPCA personnel. The site visit in October illuminated no new areas where suggestions need to be made.

It is the belief of MRI personnel that an active quality assurance program be initiated as soon as practical. This will aid the Reserve laboratory personnel and others in evaluating their performance.