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CANADA

DEPARTMENT OF MINES AND TECHNICAL SURVEYS

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 62-12

**CONDUCTIMETRIC TESTS ON SOLUTION
FROM CANADIAN COPPER REFINERS LTD.**

by

T. R. FLINT

MINERAL SCIENCES DIVISION

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CONDUCTIMETRIC TESTS ON SOLUTION
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SUMMARY OF RESULTS

Conductimetric control tests have been conducted on an acid leach filtrate solution containing 78 g/l Cu, 34.4 g/l H_2SO_4 and 12.8 g/l Te at a temperature of 65°C with a laboratory conductimetric immersion probe. The purpose of the tests was to establish if the conductimetric probe could be used to control the acid concentration in the solution. It was found that stable increments of 0.5 mV could be obtained on a 10-millivolt recorder for 0.6 g/l additions of H_2SO_4 . These increments are more than adequate for control purposes. Addition of copper sulphate in excess of 100 g/l to a 3 litre test solution had no appreciable effect on the probe output reading.

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INTRODUCTION

Following a request from Canadian Copper Refiners Limited, Montreal East, Que. in November 1961, tests have been conducted on a saturated copper sulphate solution to establish if the conductimetric immersion probe (1, 2) could be used to control the acid concentration in the Company's leach solution. The probe used was a laboratory unit, without temperature compensation, of the type described previously (2).

Comparative tests were done on pure H_2SO_4 solutions at various concentrations and on an acid leach filtrate submitted by the Company containing 78 g/l Cu, 12.8 g/l Te and 34.4 g/l H_2SO_4 . Since the leach solution was saturated with copper sulphate at room temperature, it was necessary to work with hot solutions to obtain the desired copper ion concentrations. All tests were conducted in a constant temperature bath at 65°C (149°F). Copper sulphate was added in a subsequent test to observe the effect of varying copper concentrations on the probe output. Following the Company's suggestion, it was assumed that the silver and tellurium ion concentrations will not vary substantially to affect the conductivity.

PROCEDURE AND APPARATUS

Figure 1 shows a picture of the apparatus; on the left is a Honeywell Model 17 Recorder (10 mV sensitivity) and on the right is the Oscillator and Detector unit of the system. In the foreground is a typical probe coated with a clear Tygon compound to protect the metal parts from corrosion. For these tests the probe used was coated with an epoxy potting compound reinforced with cotton tape.

Figure 2 shows a picture of the equipment layout. By means of the thermostatic bath the temperature of the solution could be held within 0.5°C and, due to the agitation, a homogeneous solution was quickly obtained after each acid addition.

A series of three tests was carried out on a synthetic CuSO_4 solution, an H_2SO_4 solution and the plant solution. Initial runs to determine the range of sensitivity of the unit were carried out with a 35 g/l H_2SO_4 solution. These tests were carried out in the following manner:-

- 1) The solution was heated in a 3 litre flask placed in the constant-temperature bath to a temperature of 65°C (149°F).
- 2) The H_2SO_4 additions were metered into the solution by a burette and the burette reading recorded on the chart.

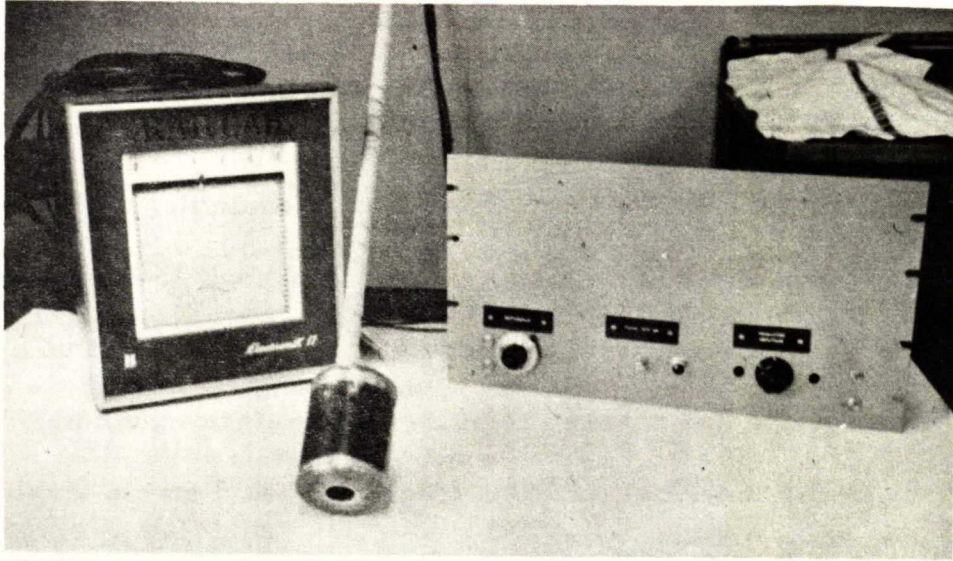


Figure 1 - Conductimetric control system

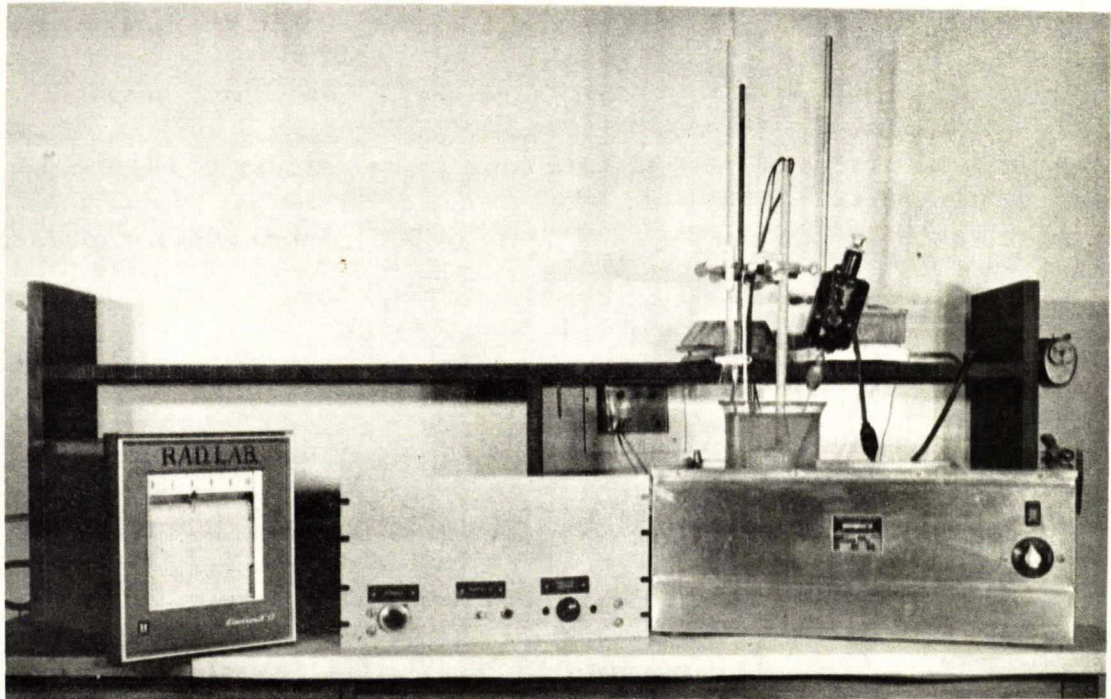


Figure 2 - Test assembly

- 3) The temperature of the solution was periodically recorded on the chart.

The H_2SO_4 solution results are shown in Figure 3. It will be noted, that there was an interval after the addition of 11.05 ml (burette reading). This interval was provided to allow the solution temperature to stabilize. It was noticed during the last addition that the temperature had changed to 66.5°C ; therefore, the unit was allowed to run until the recorder trace reached a stable level again. As can be observed, a 0.5°C change represents approximately 0.8 mV change in output.

Figure 4 shows the results obtained with the CCR acid leach solution. The initial sensitivity (2.1 ml burette reading) was noted to be quite low, 0.3 mV/ml; therefore the oscillator output amplitude was increased by 30%. From the remainder of Figure 4 it can be seen that there is a sensitivity of 0.5 mV/ml which represents 0.80 mV/g/l.

Figure 5 shows the results obtained from a synthetic solution to which crystalline $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ was added. This solution was prepared as an approximation to the plant solution to conserve the CCR leach solution and to confirm preliminary results with the final solution from the previous test. There was a small initial deflection followed by a return to the normal reading. Some minor variation attributable to incomplete solution of the CuSO_4 appeared. However, as would be expected from the low dissociation of CuSO_4



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Sulphuric Acid Solution

3000 ml water

58.5 ml H₂SO₄

Acid Sp. Gr. 1.84

Purity 0.94

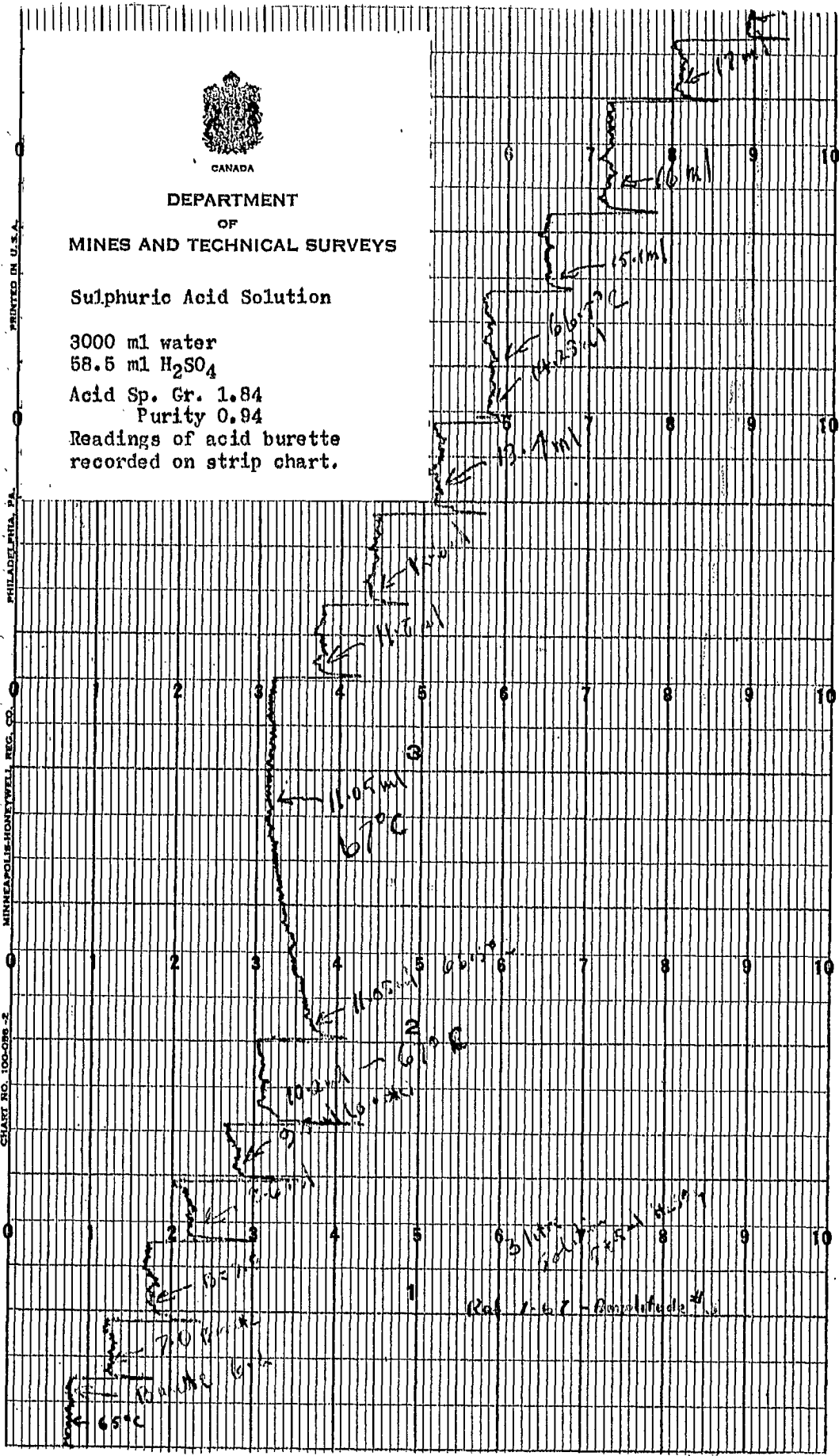
Readings of acid burette recorded on strip chart.

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↑
TIME

mV →

Figure 3 - Recorder trace - H₂SO₄ solution
H₂SO₄ additions



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Canadian Copper Refiners Ltd.

Acid leach filtrate
3000 ml Plant Solution 65°C
H₂SO₄ Additions
Acid Sp. Gr. 1.84
Purity 0.94

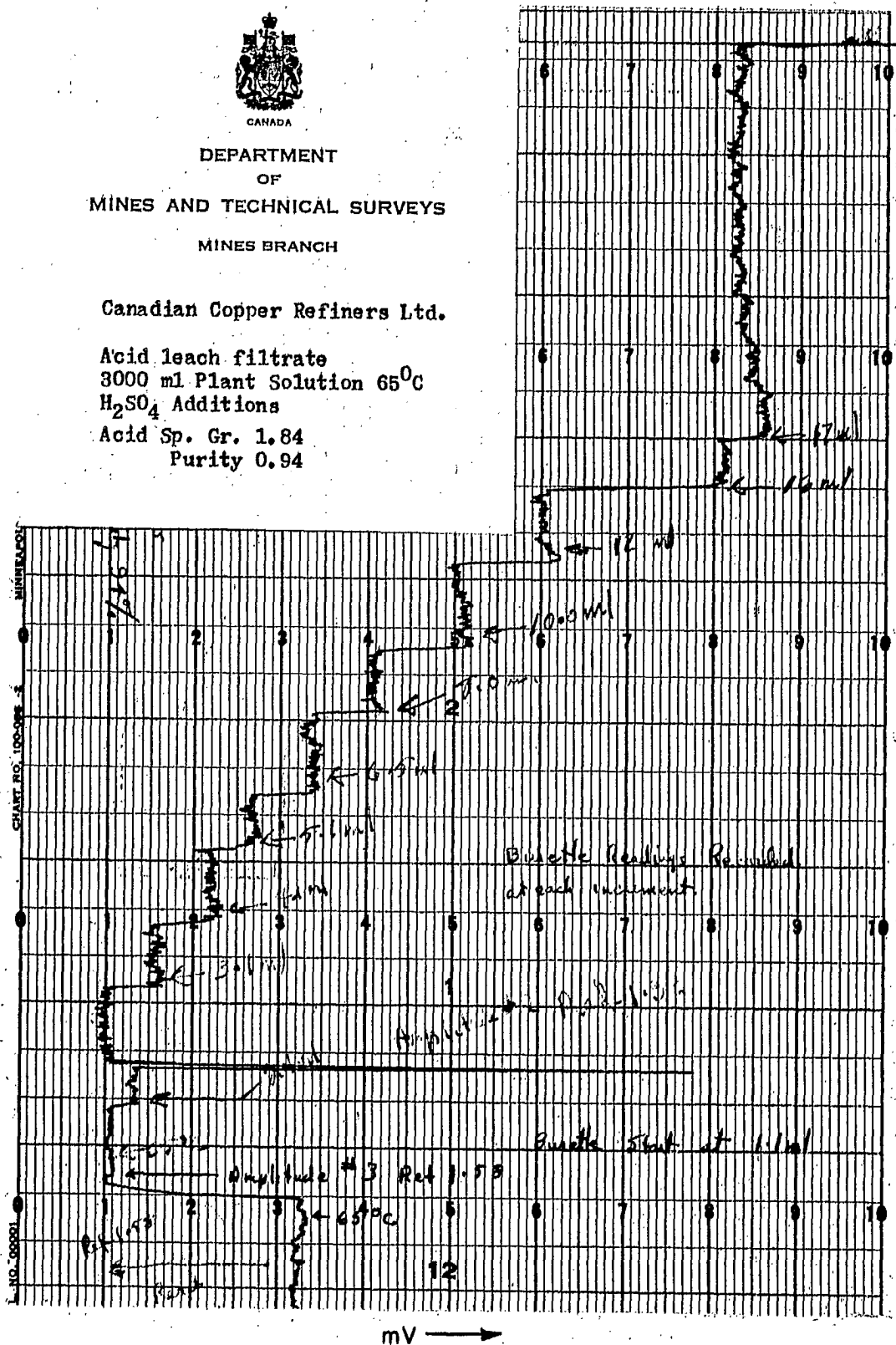
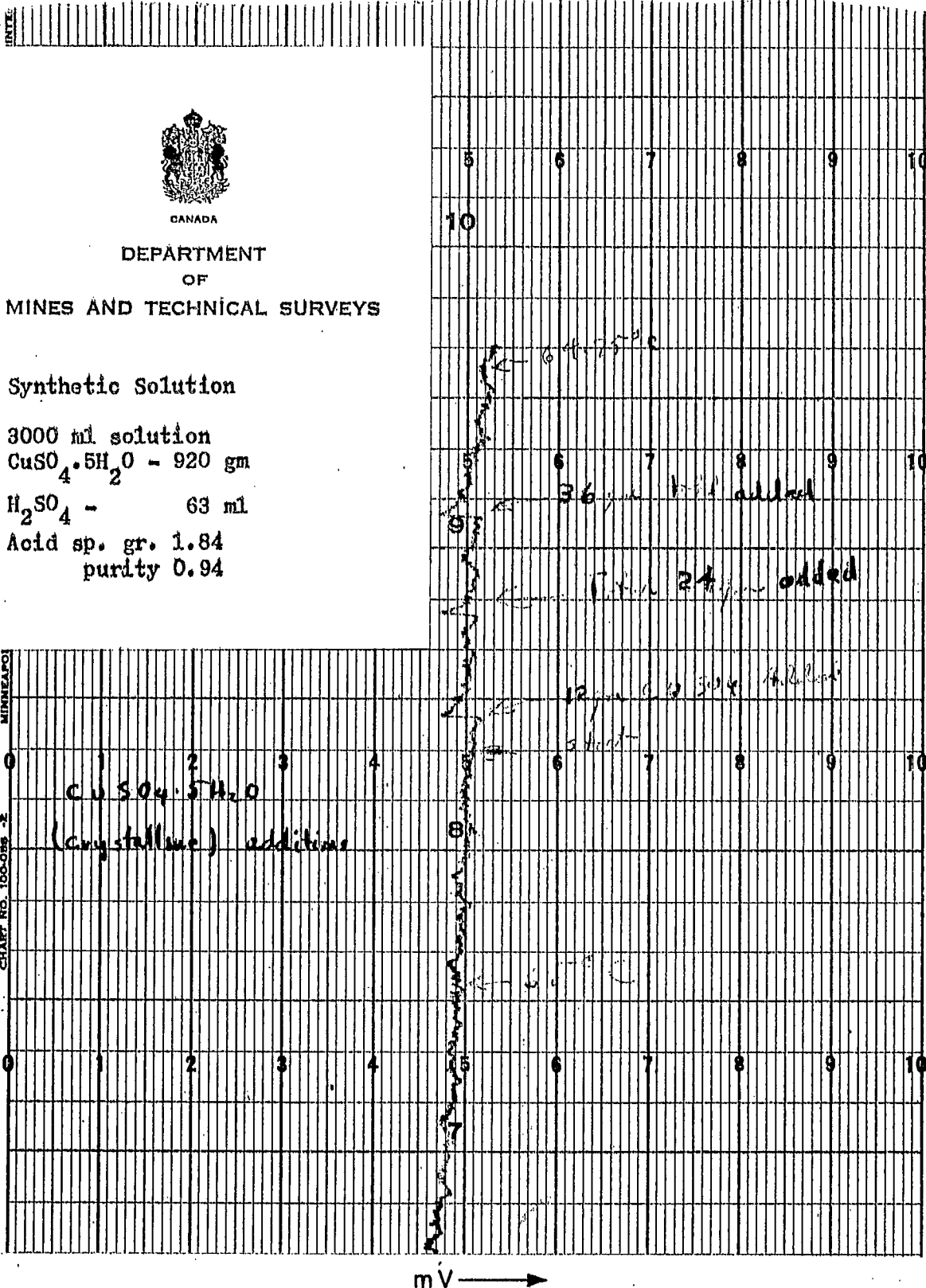


Figure 4 - Recorder trace - Plant filtrate solution
H₂SO₄ additions.




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Synthetic Solution

3000 ml solution
 $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ - 920 gm
 H_2SO_4 - 63 ml
 Acid sp. gr. 1.84
 purity 0.94

$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$
 (crystalline) additions

Figure 5 - Recorder trace - CuSO_4 solution

CuSO_4 additions

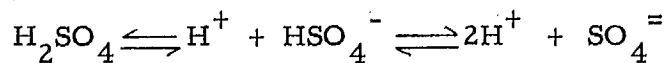
in the presence of strong $\text{SO}_4^{=}$ ion, there was little effect upon the conductivity of the solution.

As can be seen from Figure 5, a synthetic solution also does not show any long-term change in conductivity as CuSO_4 is added. It will be noted that there was a progressive drop in temperature of 0.25°C during the test which accounts for the progressive increase in solution conductivity; however $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ additions introduce only minor perturbances in the recorder trace.

DISCUSSION

It is evident from the tests that successful acid leach control can be accomplished in this process by conductimetric methods, provided the temperature can be kept constant. Otherwise temperature compensation must be incorporated in the probe (2).

It will be noted that there is a negative temperature coefficient in this solution which is probably attributable to the following competing reactions:



At any given temperature and concentration there will be an equilibrium between HSO_4^- and $\text{SO}_4^{=}$ ions; within the range of temperatures of interest here, any increase in temperature results in a decrease in conductivity as the increase in thermal energy favours the existence of HSO_4^- ions.

Because essentially no free Cu^{++} ions exist in solution under strong SO_4^{--} conditions, gross additions of CuSO_4 will have no appreciable effect upon conductivity.

The practicality of this method of acid concentration control appears to be good. As can be seen, the traces indicate that with equipment specifically designed for this solution system much greater differential sensitivity could be obtained. Since the apparatus used was not temperature compensated, the results were subject to some thermal drift. It was merely a matter of convenience that compensation was omitted in these tests, although satisfactory compensation has been developed for other processes. The results with the equipment as it stands provide a sensitivity of 0.8 mV/g/l with a thermal coefficient of -1.6 mV/deg C. Since the sensitivity of this system can be adjusted over a wide range and since the thermal coefficient can be reduced several orders of magnitude by proper temperature compensation, it appears that this system will provide satisfactory control for variations in acid concentrations of the order of 0.25 g/l, if care is taken in the design and installation of the unit.

REFERENCES

1. G.G. Eichholz and A.H. Bettens, Conductimetric Measurement and Control of Acid Concentration in Leach Pulps, Trans. C.I.M.M., 63, 625 (1960). Mines Branch Technical Bulletin TB 17, 1960.
2. G.G. Eichholz and T.R. Flint, Conductimetric Control of Alkaline Leach Liquors, Mines Branch Technical Bulletin TB 27, 1961.

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