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EVALUATION OF A CONTINUOUS ^{14}C , ^{129}I , and ^{85}Kr MONITOR UNDER DISSOLVER OFF-GAS CONDITIONS

SEPTEMBER 1983

Idaho Falls, Idaho 83401

~~EX~~ON NUCLEAR IDAHO COMPANY, Inc.

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EVALUATION OF A CONTINUOUS ^{14}C , ^{129}I , and ^{85}Kr MONITOR
UNDER DISSOLVER OFF-GAS CONDITIONS

by

S. J. Fernandez
G. J. McManus
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October 1983

EXXON NUCLEAR IDAHO COMPANY, INC.

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DEPARTMENT OF ENERGY
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ABSTRACT

A previously developed ^{129}I , ^{14}C , and ^{85}Kr stack monitor was evaluated for use on dissolver off-gas streams. The effect of higher concentrations of H_2 , Hg (a dissolution catalyst), and Cd (a common neutron poison) was measured in a series of laboratory tests. No significant effect on the sensitivity of the monitor was observed due to the presence of higher H_2 concentrations; but the variability did increase, probably due to condensation. Both Cd and Hg significantly reduced the sensitivity and increased the noise level of the monitor. Therefore, the ^{129}I monitor, although suitable for stack monitoring, was considered unsuitable for dissolver off-gas streams containing high concentrations of Hg and Cd .

SUMMARY

The continuous monitoring of gaseous ^3H , ^{14}C , ^{129}I and ^{85}Kr releases from nuclear fuel reprocessing plants is important from regulatory and process control viewpoints. A continuous ^3H , ^{14}C , ^{129}I and ^{85}Kr monitor was developed¹ that used selective permeation to separate each radionuclide and on-line conversion of I_2 to CH_3I . The methyl iodide conversion used a free radical reaction on a catalytic surface that is potentially sensitive to H_2 (through side reactions forming HI), Hg and Cd (through catalyst poisoning).

This work evaluated the effect of Hg , Cd , and H_2 because these substances are commonly found in dissolver off-gases. The approach was to measure the sensitivity and reliability of the monitor to a constant source of $\text{CH}_3^{129}\text{I}$ as the levels of H_2 , Hg and Cd were varied in a 2^3 factorial design.

The major conclusions from this work were:

- 1) H_2 had no significant effect on the sensitivity of the monitor but had a significant effect on the variability of the monitor;
- 2) The presence of Hg reduced the response of the monitor to 30% of its original value and significantly increased the variability of the monitor;
- 3) The presence of H_2 significantly reduced the Hg effect, perhaps by reducing the Hg to the easily volatilized elemental form;
- 4) The presence of Cd reduced the response of the monitor to a statistically insignificant level.

From these results, the ^{129}I monitor, while suitable for stack monitoring, was shown to be unsuitable for dissolver off-gas streams containing high concentrations Hg and Cd .

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I. INTRODUCTION

Tritium, carbon-14, iodine-129 and krypton-85 are among the most environmentally significant radioisotopes emitted from fuel reprocessing and waste solidification facilities. Also, they are among the most difficult to measure in realtime. This difficulty derives from the fact these nuclides either decay with the emission of only low-energy beta particles (^3H and ^{14}C), or with the emission of weak X-rays and γ -rays of low absolute abundance (^{85}Kr). In addition, tritium, carbon-14, and iodine-129 may simultaneously exist in more than one gaseous chemical form.

Despite these difficulties, nuclear fuel reprocessing and waste solidification facilities are required to monitor ^3H , ^{14}C , ^{85}Kr , and ^{129}I releases to the environment. An off-gas monitor used to measure these nuclides in stack gases has been developed at the ICPP.¹

A functional diagram of this monitor is shown in Figure 1. The filtered sample stream is mixed with methane and drawn through a Pt-Pd on alumina oxidizing catalyst (catalytic oxidizer) maintained at 500°C . This catalytic oxidizer quantitatively converts HT to HTO and removes oxygen from the sample stream by oxidation of the methane. The sample stream is then split, one-half is directed to the continuous ^3H monitor and the other is directed to the continuous ^{129}I , ^{85}Kr and ^{14}C monitor.

The continuous ^3H monitor consists of a selective permeation device (described in section 1.1) that separates the HTO from the sample stream and a 20-L ionization chamber that continuously measures the HTO concentration.

The continuous ^{129}I , ^{85}Kr , and ^{14}C monitor first converts any I_2 in the sample stream to CH_3I . Stable I_2 carrier and $(\text{CH}_3)_3\text{Bi}$ (source of free methyl radicals) are added to the sample stream and the conversion reaction proceeds over a Rh on alumina catalyst maintained at 500°C . The ^{85}Kr is separated from the $\text{CH}_3^{129}\text{I}$ and $^{14}\text{CO}_2$ by a selective permeation device (described in the next section) and monitored with a surface barrier detector. The $\text{CH}_3^{129}\text{I}$ and $^{14}\text{CO}_2$ are filtered to eliminate potential ^{125}Sb interferences. The $\text{CH}_3^{129}\text{I}$ is collected on a charcoal bed and the 29 KeV X-ray measured with a 1mm by 25mm NaI(Tl) detector (FIDLER). The $^{14}\text{CO}_2$ sample is dried with a silica gel bed to eliminate any traces of HTO before the ^{14}C concentration is measured with an ionization chamber. The selective permeation devices are described in the next section.

1.1 Selective Permeation

The ability of some polymers to separate permanent gases has been known for many years.² The law governing the permeation of a gaseous constituent across a membrane is:

$$J = \frac{P \Delta p}{h} \quad (1)$$

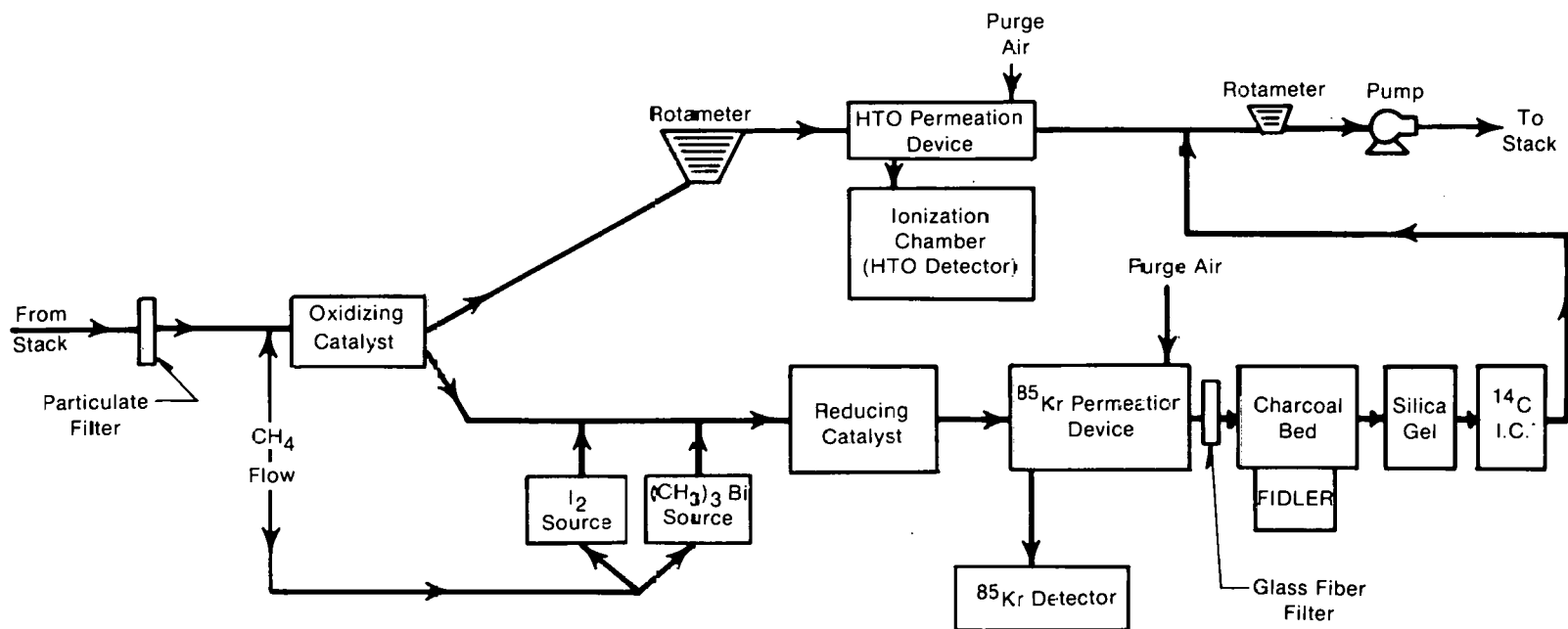


Figure 1. Continuous 3H , ^{14}C , ^{129}I , and ^{85}Kr Monitor

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where: J is the flux of a gaseous constituent across the membrane;
 P is the permeability of the polymer to the diffusing gas;
 Δp is the difference in bulk partial pressure of the diffusing gas across the membrane;
 h is the thickness of the membrane.

From Equation 1 it can be inferred that the flux of a gas through a given membrane can be predicted if the membrane's permeability is known. Conversely, if the permeability of several gases (I_2 , CH_3I , Xe , Kr) is known, their separation factors can be derived for a given membrane.

Using the equations derived by Treece, et. al.³, these factors can be calculated for a tube and shell membrane configuration, a configuration easily adapted to continuous off-gas monitoring. If we define K (the separation factor) as the ratio of the partial pressure of the diffusing gas in the product to the partial pressure of the diffusing gas in the purge, then:

$$K = \frac{\Phi \ln(b/a)}{2 \pi L P p_t} + 1 \quad (2)$$

where: Φ is the volumetric flowrate of the purge stream;
 L is the length of the polymeric tubing;
 p_t is the total pressure of the product stream;
 b is the outer diameter of the tubing;
 a is the inner diameter of the tubing.

From Equation 2, permeabilities (and thereby separation factors) can be calculated if the partial pressures in the purge and product streams are measured. Conversely, once the permeabilities are known, Equation 2 can be used to design a device to achieve the desired separation factors.

The tritium selective permeation device used a perfluorosulfonic acid polymer (Nafion®, Dupont). The permeation device contained 100 tubes. Each tube was 1.21 m long with inside and outside diameters of 0.05 cm and 0.10 cm, respectively. The permeability of Nafion for water is $1.9 \times 10^{-5} \text{ cm}^3 \text{ (STP)/(cm} \cdot \text{cmHg} \cdot \text{sec)}$, but only $2 \times 10^{-10} \text{ cm}^3 \text{ (STP)/(cm} \cdot \text{cmHg} \cdot \text{sec)}$ for ^{85}Kr . Therefore, a separation factor $> 2 \times 10^4$ for tritium from ^{85}Kr was achieved.

The ^{85}Kr selective permeation device used a silicone rubber, Silastic® (Dow Corning). Separation factors from $CH_3^{129}\text{I}$ of 100 and from ^{14}C of 10^4 were demonstrated.

1.2 Dissolver Off-Gas Monitoring

The increasing desirability of reducing ^{129}I emissions to the environment has increased the justification for continuously monitoring ^{129}I in dissolver off-gases upstream of iodine abatement systems. To successfully measure ^{129}I (in the presence of ^{14}C and ^{85}Kr) the monitor must tolerate higher levels of H_2 (up to >99%) Hg (a common dissolution catalyst) and Cd (a common neutron poison). This work evaluated the effect of H_2 , Hg, and Cd on the ^{129}I portion of the previously described monitor.

The approach used to evaluate the effect of H_2 , Hg and Cd was a 2^3 factorial design. A 2^3 factorial design examines the response of the monitor as each factor (H_2 , Hg and Cd) is varied between two levels. The levels used in this experiment were H_2 (0, >50%); Hg (0, 100 μg); Cd (0, 100 μg). The effect of each factor on both the sensitivity and variability of the monitor was examined. The detailed experimental procedure is presented in the next section.

II. EXPERIMENTAL

The experimental apparatus shown in Figure 2 was used in this evaluation. Air was sampled at a flowrate of 160 cm³/min and combined with 270 cm³/min of CH₄ (or H₂ if a H₂ test was being performed). After the catalytic oxidizer, a constant stream of CH₃ ¹²⁵I was introduced by passing a 15 cm³/min flow of CH₄ (or H₂) over a traced CH₃ ¹²⁵I permeation tube. After the CH₃ ¹²⁵I conversion catalyst, the CH₃ ¹²⁵I was carried through a ⁸⁵Kr selective permeation device. The selective permeation device was calibrated using a constant I₂ permeation device that generated 70 pCi/min ¹²⁵I₂. A calibration factor of 7.1 x 10⁶ cps (μCi/cm³)-¹d-¹ was measured assuming a 160 cm³/min sampling flowrate. This calibration constant was maintained within 15% for 33 hours, demonstrating the reliability of the permeation device. The counter-current purge gas was supplied by a small compressor. The CH₃ ¹²⁵I penetrating the selective permeation device was collected by a 55 cm³ TEDA-impregnated charcoal bed and the 27 KeV X-ray measured with a 1mm by 25mm NaI(Tl) detector (FIDLER). The observed countrate was recorded with an ND60 programmable multichannel analyzer.

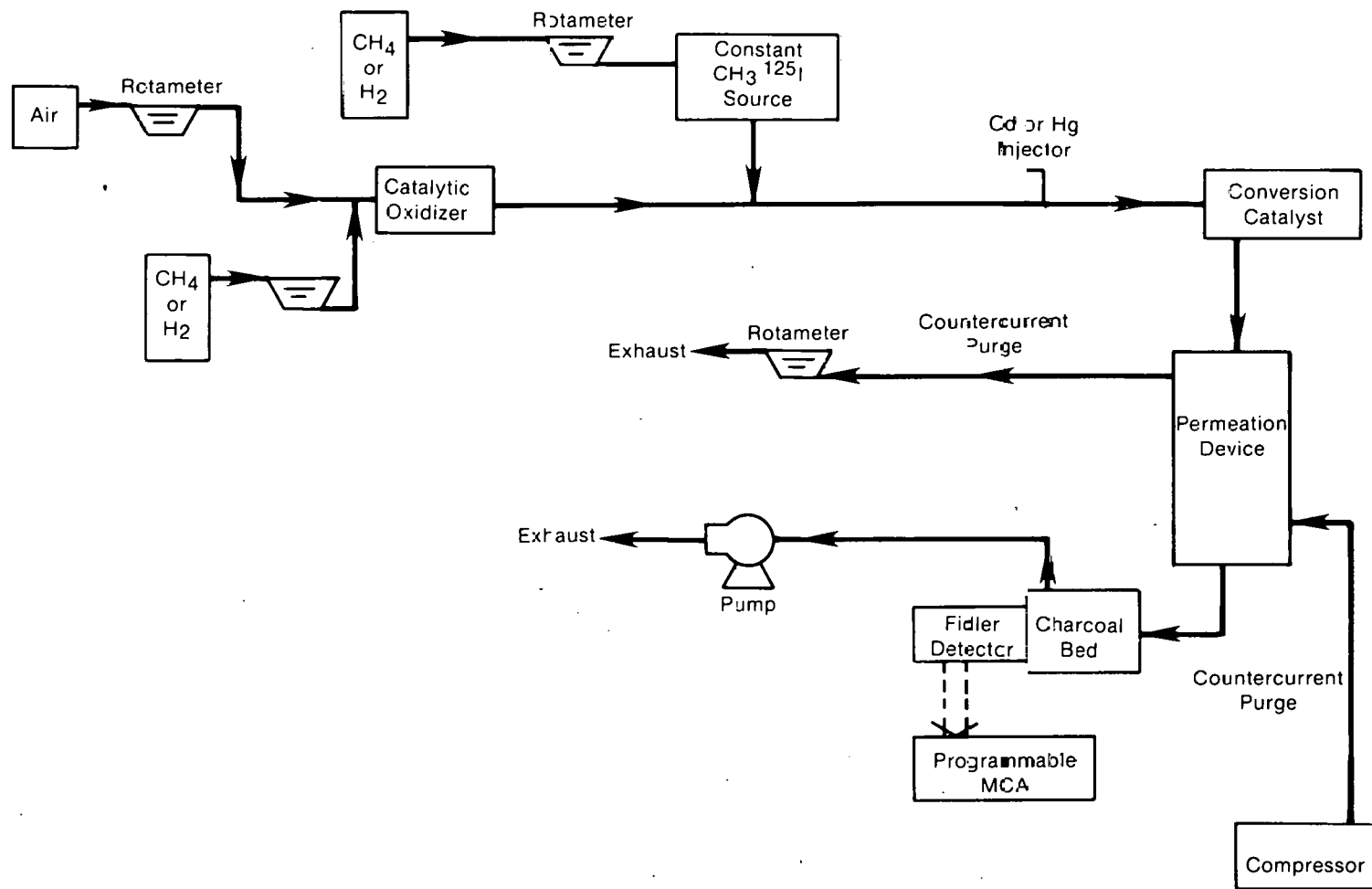
The Cd or Hg tests were preceded by the injection of 100 μL of 1 mg/mL Hg(NO₃)₂ or CdCl₂ solution into the sample stream immediately upstream of the heated conversion catalyst bed. This injection method simulated the aerosol deposition one would encounter when sampling dissolver off-gas streams. The 100 μg level is typical of the quantity collected after a few months sampling.

The 2³ factorial experimental design is shown in Table I. Each test condition was performed in duplicate for a total of 16 tests. Each test consisted of between 4 and 9 individual counts collected over time periods between 70 minutes and 66 hours. The countrates were then plotted and the slopes and linear correlation coefficients (r) determined. The slope of the countrate vs time plot is directly

TABLE I

2³ EXPERIMENTAL DESIGN

Test Number	H ₂ Concentration (%)	Hg Added (μg)	Cd Added (μg)
1-2	0	0	0
3-4	64	0	0
5-6	0	100	0
7-8	64	100	0
9-10	0	0	100
11-12	64	0	100
13-14	0	100	100
15-16	64	100	100



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Figure 2. Modified Continuous 129I Monitor

proportional to the sensitivity (response per unit ^{125}I activity). The quantity $(1-r^2)$ is directly proportional to the variability of the response around its average value. Therefore, the effect of H_2 , Hg and Cd on average response and variability of response were determined. The statistical analysis of 2^3 factorial designs followed that detailed in standard statistics textbooks.⁴

III. RESULTS AND DISCUSSION

The results of the 16 tests are summarized in Table II. The sensitivities were normalized to the response observed in the absence of H₂, Hg and Cd. The plots of each test are presented in Appendix I and the counting data is tabulated in Appendix II.

The effect of each factor was calculated in the manner of Walpole and Myers⁴ (an example calculation is shown in Appendix III) and the results are summarized in Table III. Any effect on response greater than 10% and any effect on variability greater than 5% was considered statistically significant.

TABLE II
RESULTS OF 2³ FACTORIAL EXPERIMENT

Test Number	H ₂ Concentration (%)	Hg Content (μg)	Cd Content (μg)	Sensitivity (% of base case)	Variability (1-r ²) x 100%
1	0	0	0	91	0.67
2	0	0	0	109	10
3	64	0	0	51	0.25
4	64	0	0	110	10
5	0	100	0	1.1	16
6	0	100	0	17	32
7	64	100	0	34	21
8	64	100	0	30	10
9	0	0	100	0.23	21
10	0	0	100	-1.7	1
11	64	0	100	0.13	55
12	64	0	100	-5.0	82
13	0	100	100	18.7	25
14	0	100	100	18.3	55
15	64	100	100	0.48	98
16	64	100	100	5.7	88

3.1 Effects on Response

As seen in Table III, H₂ had no statistically significant effect on response, but Hg and Cd decreased the response of the monitor by significant amounts. The large Hg effect (-29%) and large Cd effect (-51%) combined with the large Hg + Cd interaction (+41%) suggests that the Hg effect and Cd effect may be underestimated as an artifact of the manner used to calculate the effects. For example, if both the Hg effect and Cd effect were -75%, the sum of two effects would be -150%. Because the maximum effect observable in a test containing Hg and Cd is -100%, the Hg + Cd interaction is (-100)-(-75)-(-75) or +50%, even in the absence of a physical interaction between the Hg and Cd. In addition, this "virtual" interaction reduces the actual Hg effect and Cd effect when the

TABLE III

EFFECT OF H₂, Hg AND Cd ON THE CONTINUOUS OFF-GAS MONITOR

<u>Factor</u>	<u>Effect on Response (%)</u>	<u>Effect on Variability (1-r²)x100%</u>
H ₂	-3	+26
Hg	-29	+20
Cd	-51	+40
H ₂ + Hg interaction	+7	-2
H ₂ + Cd interaction	-7	+15
Hg + Cd interaction	+41	+3
H ₂ + Hg + Cd interaction	-7	0

Hg + Cd test is averaged with the other runs (see calculation in Appendix III). Therefore, the data in Table II were reanalyzed as a pair of 2² factorial experiments (H₂ + Hg; H₂ + Cd). The results are shown in Tables IV and V.

From Table IV it is seen that the Hg effect was -70%; from Table V the Cd effect was -89%. Therefore, the predicted observed response to the presence of both Hg and Cd is (-70-89+41) or -118%. This is in good agreement with test 15 and 16 in Table II and in better agreement with tests 13 and 14 than the effects calculated from the 2³ factorial results.

The H₂+Hg interaction (Table IV) is significant and may result from reduction of Hg(II) to Hg(0) by the H₂ with subsequent volatilization of the elemental mercury. In any event, the H₂ + Hg interaction appears to significantly ameliorate the Hg effect.

TABLE IV

RESULTS OF H₂ + Hg 2² FACTORIAL EXPERIMENT

<u>Factor</u>	<u>Effect on Response (%)</u>	<u>Effect on Variability (1-r²)x100%</u>
H ₂	+2	+4
Hg	-70	+15
H ₂ + Hg interaction	+21	-4

TABLE V
RESULTS OF H₂ + Cd 2² FACTORIAL EXPERIMENT

<u>Factor</u>	<u>Effect on Response (%)</u>	<u>Effect on Variability (1-r²)x100%</u>
H ₂	-10	+29
Cd	-89	+35
Cd + H ₂ interaction	+9	+29

3.2 Effects on Variability

From Tables IV and V, it is seen that Hg, Cd, and the Cd + Hg interaction had significant adverse effects on the variability of the monitor. The Hg and Cd effects are not unexpected because counting statistics become more important when the response of the monitor is reduced. The significant H₂ effect may be due to condensation within the charcoal bed from the H₂O formed in the catalytic oxidizer. Although the ⁸⁵Kr selective permeation device is an efficient dryer, condensation was observed during all H₂ tests.

IV. CONCLUSIONS

The major conclusions drawn from this work were:

- 1) The presence of H_2 had no statistically significant effect on the response of the monitor;
- 2) The presence of Hg reduced the response of the monitor to 30% of its original value;
- 3) The presence of Cd reduce the response of the monitor to a barely significant level;
- 4) The presence of H_2 reduced the response loss due to Hg, perhaps by reducing the Hg to the easily volatilized elemental form;
- 5) The variability of the monitor was increased by the presence of Cd and Hg, perhaps because counting statistics become increasingly important as the monitor's response decreased;
- 6) The variability of the monitor was increased by the presence of H_2 , perhaps from condensation of the H_2O of combustion.

Based on these results, the ^{129}I monitor, although suitable for stack monitoring, was considered unsuitable for use in dissolver off-gas streams containing high concentrations of Cd and Hg.

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3. L. C. Treece, R. M. Fedler, and J. K. Ferrel, "Polymeric Interfaces for Continuous SO₂ Monitoring in Process and Power Plant Stacks", Environ Sci. Techn., 10, 457 (1976).
4. R. E. Walpole and R. H. Myers, Probability and Statistics for Engineers and Scientists, McMillan Co., New York, 1968.

APPENDIX I

The following figures are plots of the 16 runs represented in Table II.

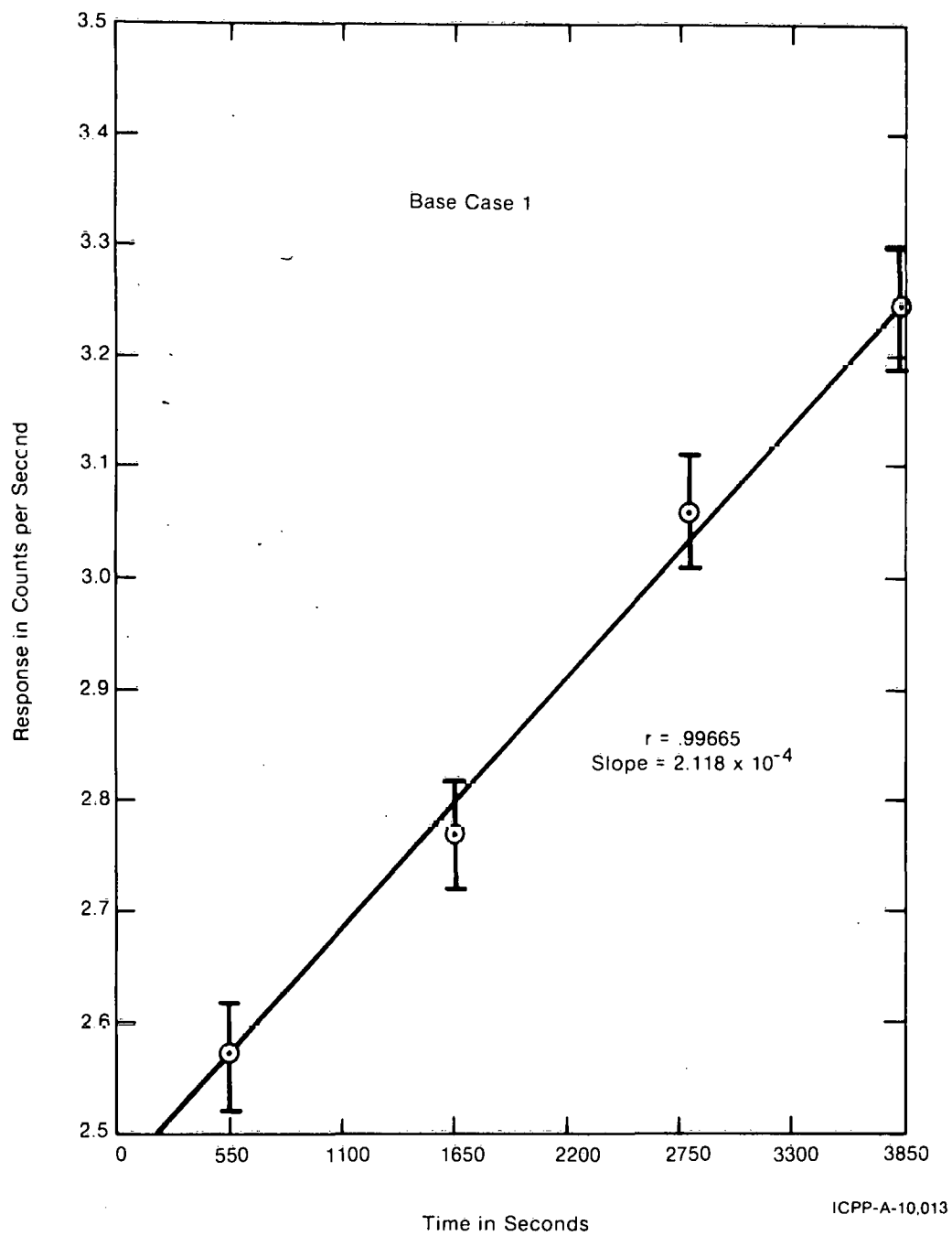
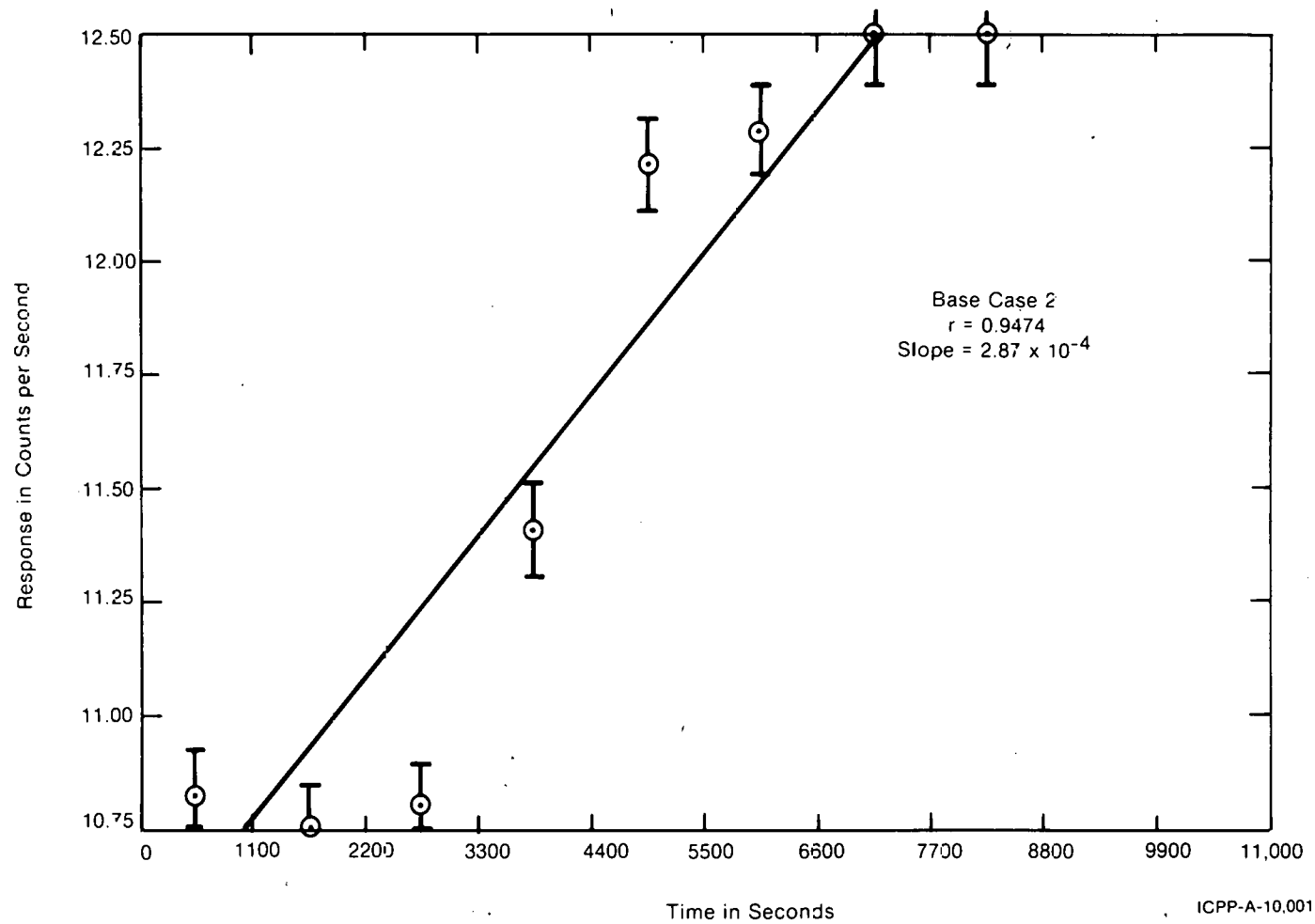


Figure A-1. Response vs Time - Test 1.

Figure A-2. Response vs Time - Test 2



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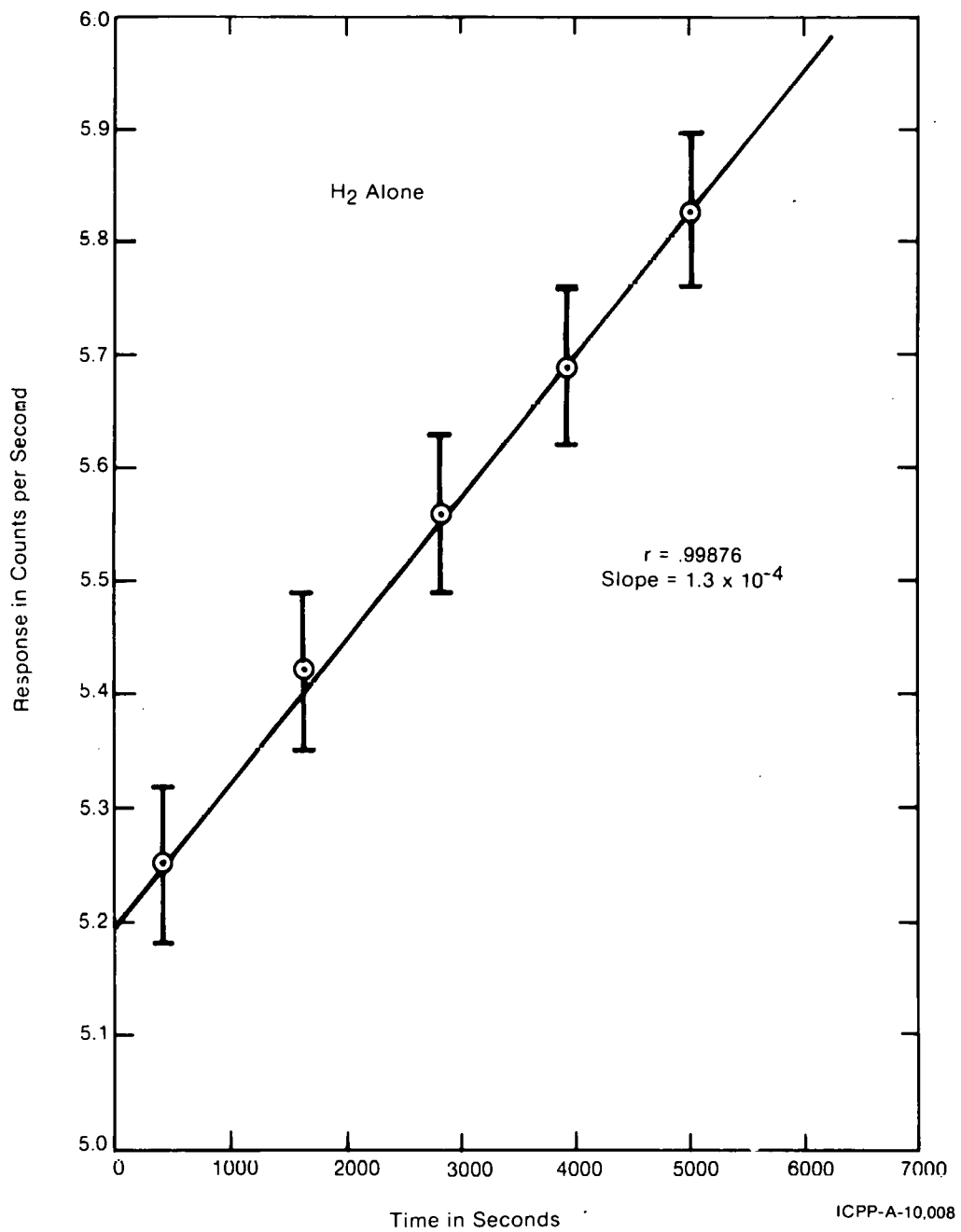


Figure A-3. Response vs Time - Test 3.

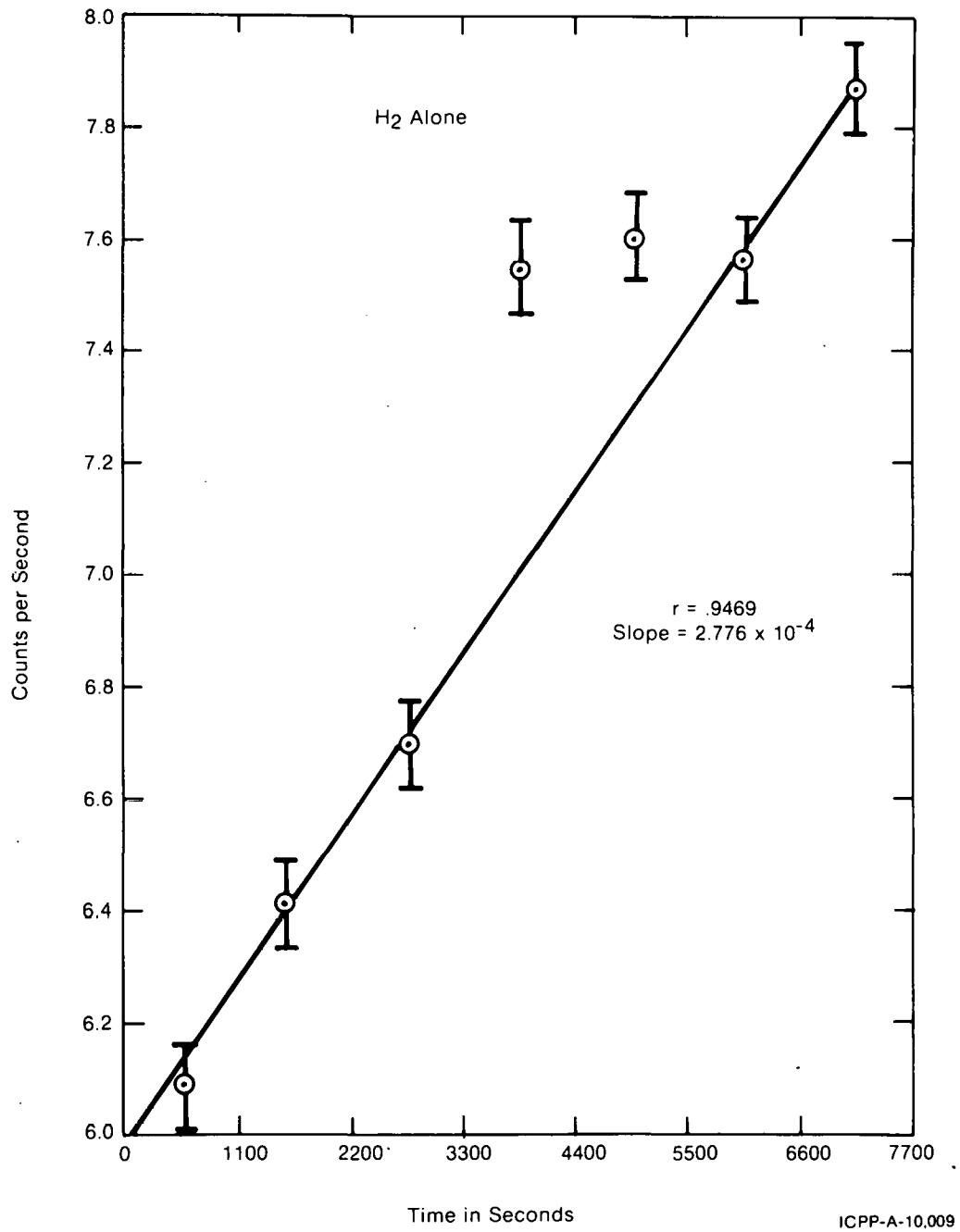


Figure A-4. Response vs Time - Test 4.

Figure A-5. Response vs Time - Test 5

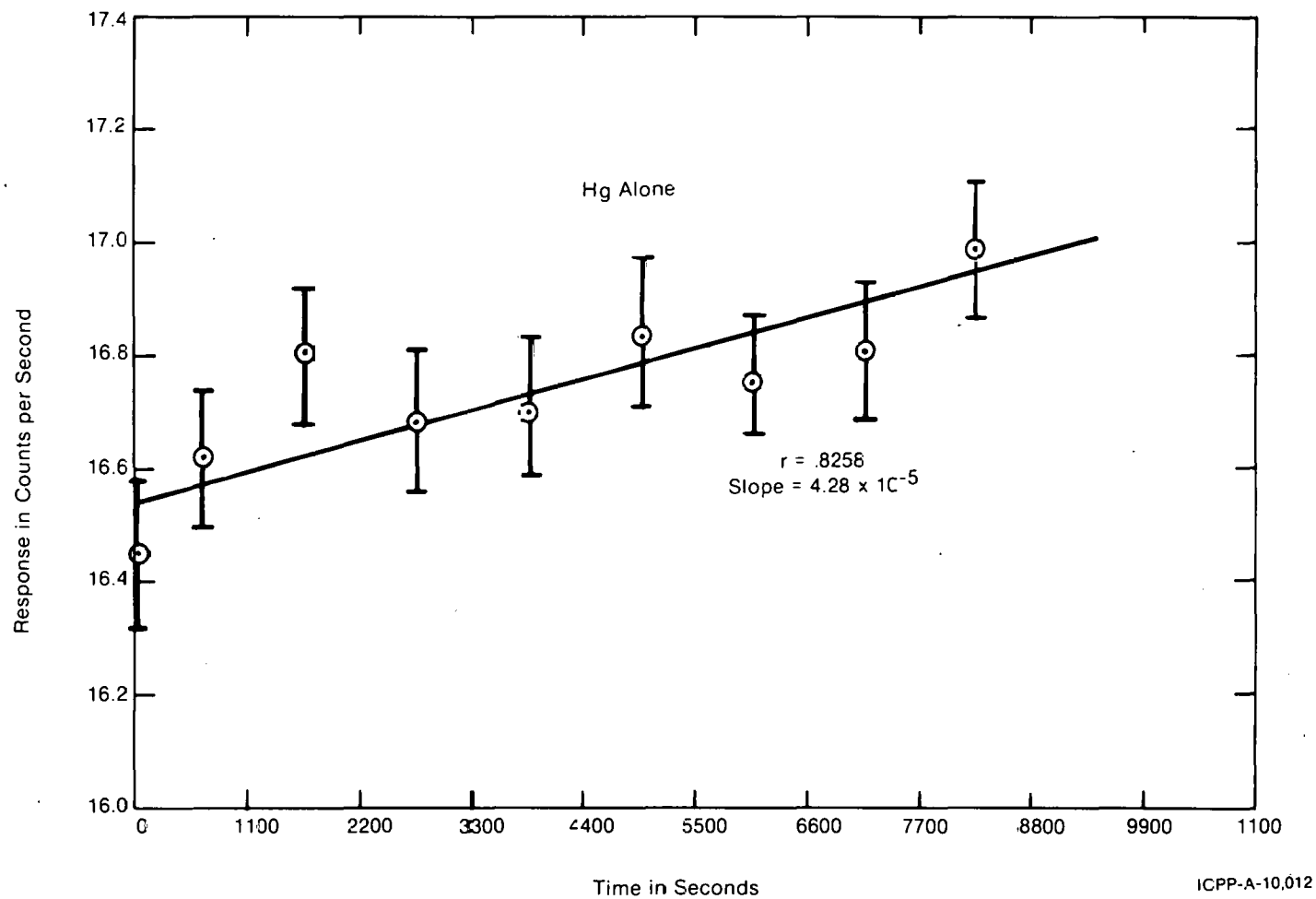
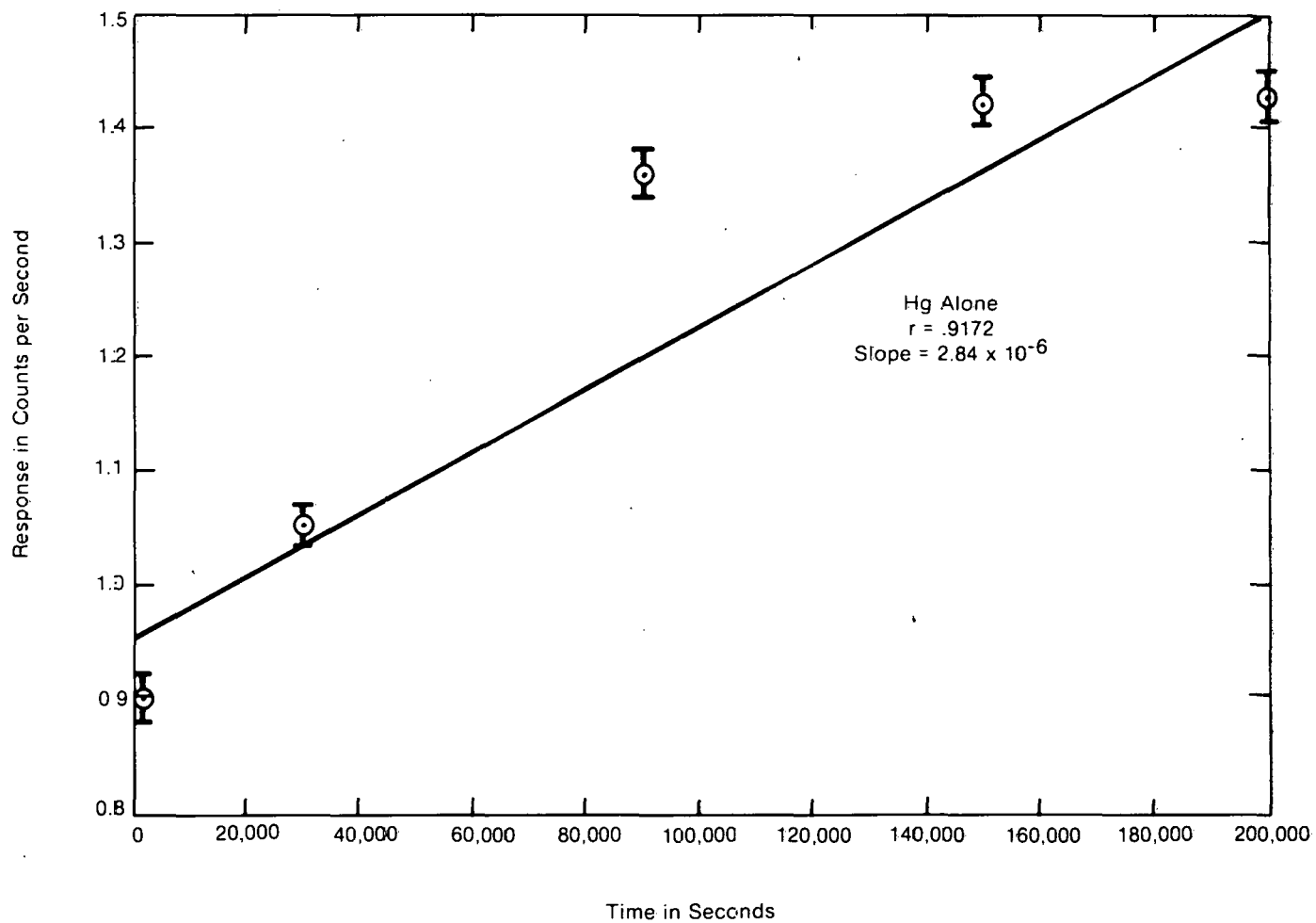


Figure A-6. Response vs Time - Test 6.



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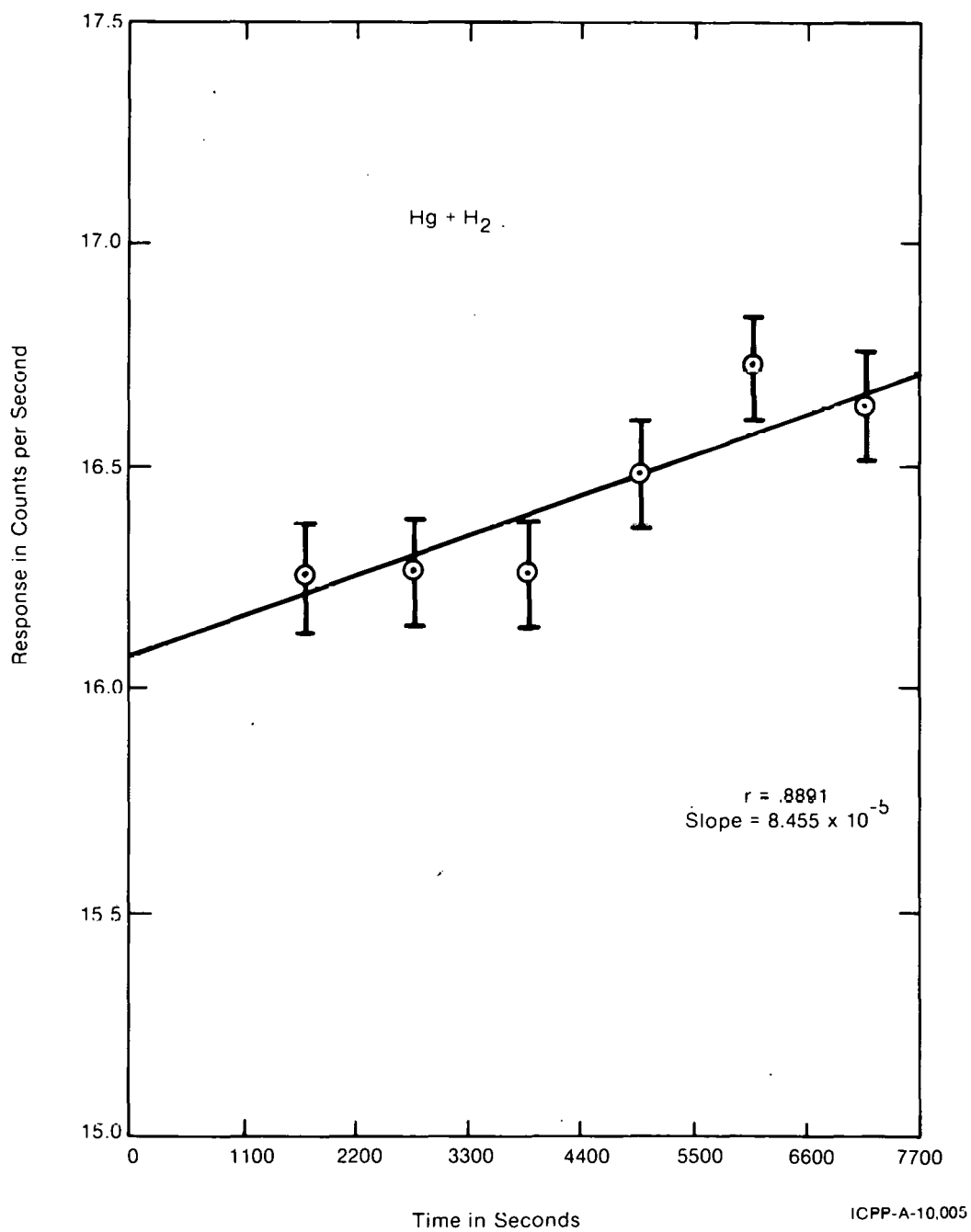


Figure A-7. Response vs Time - Test 7.

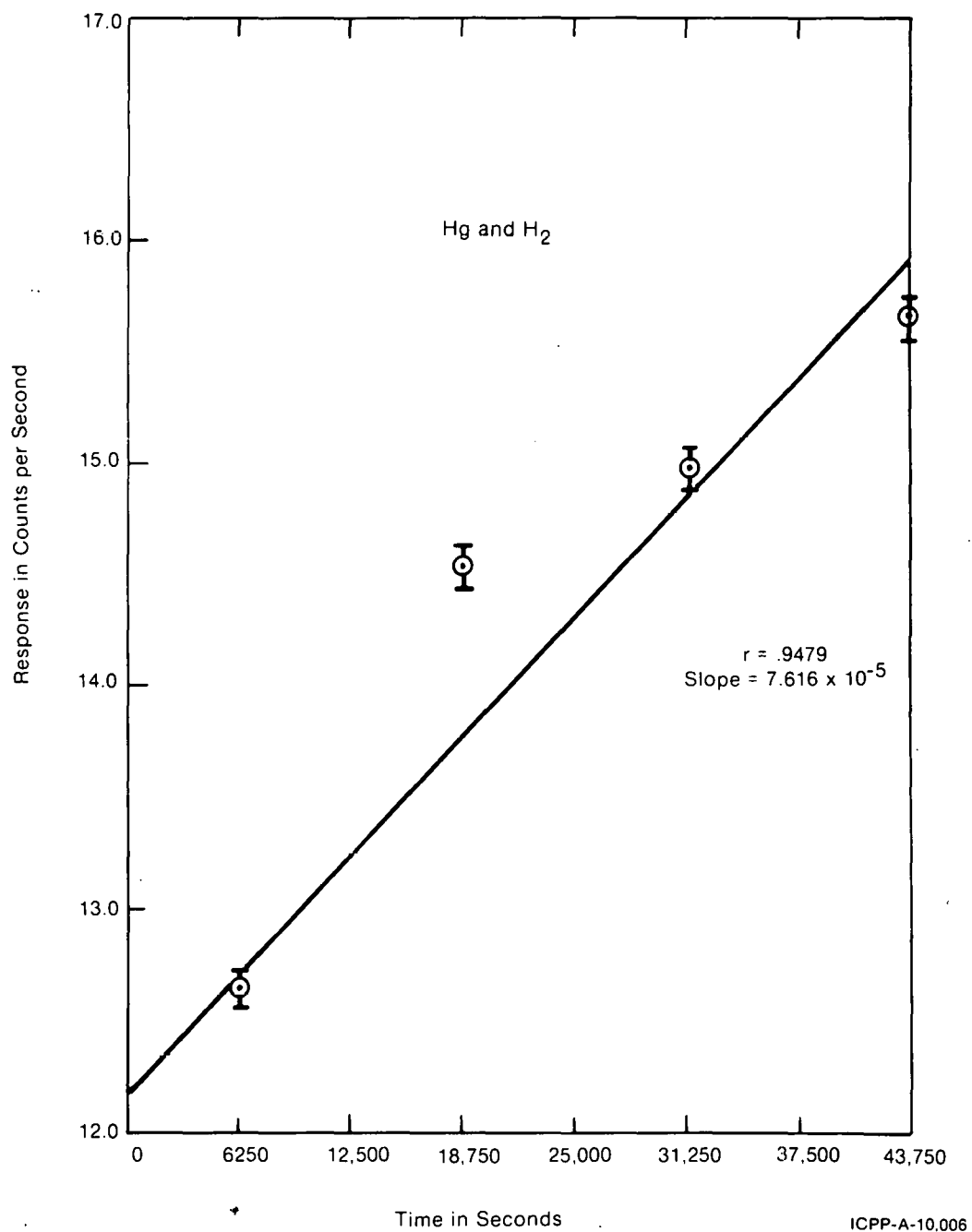
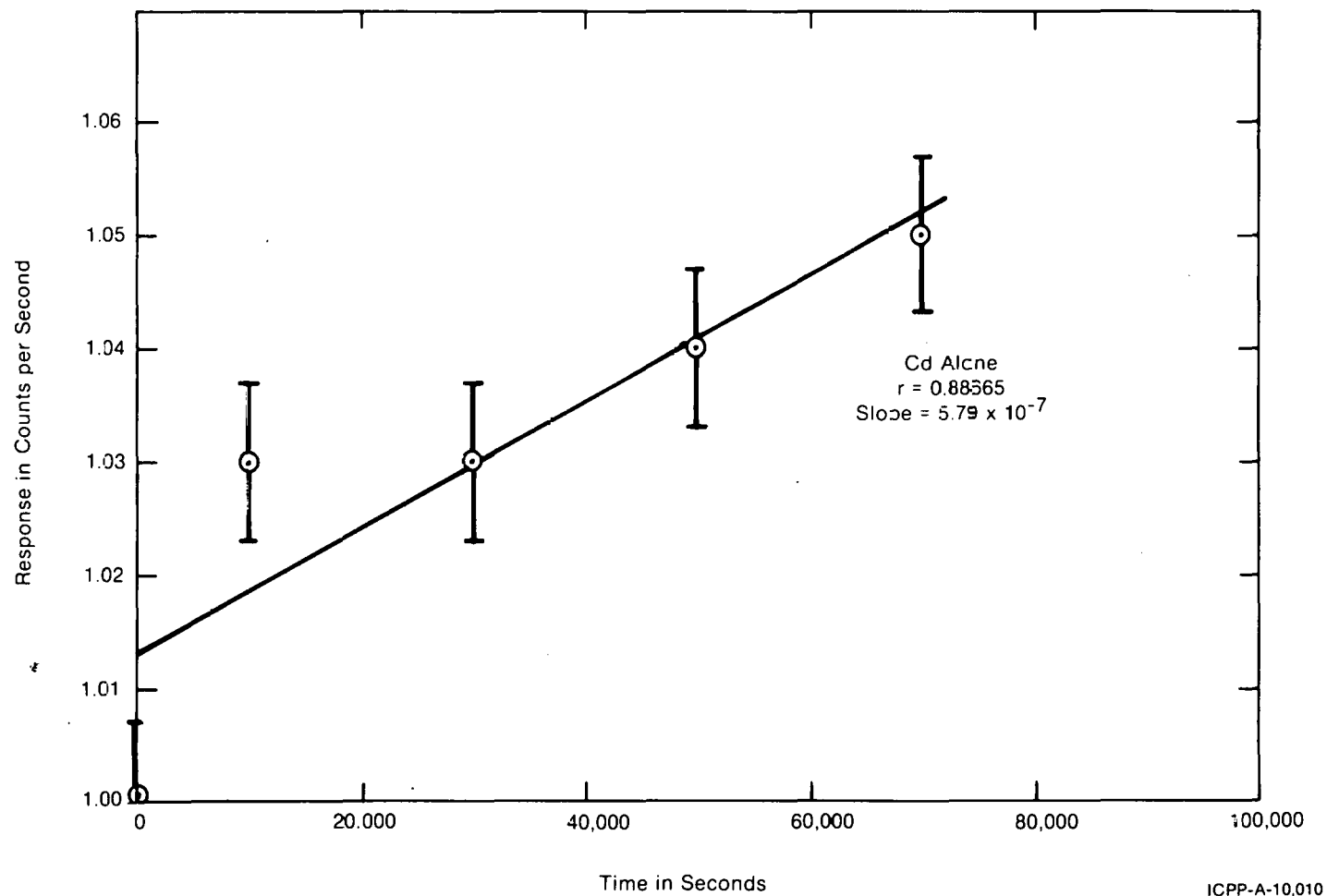


Figure A-8. Response vs Time - Test 8.

Figure A-9. Response vs Time - Test 9



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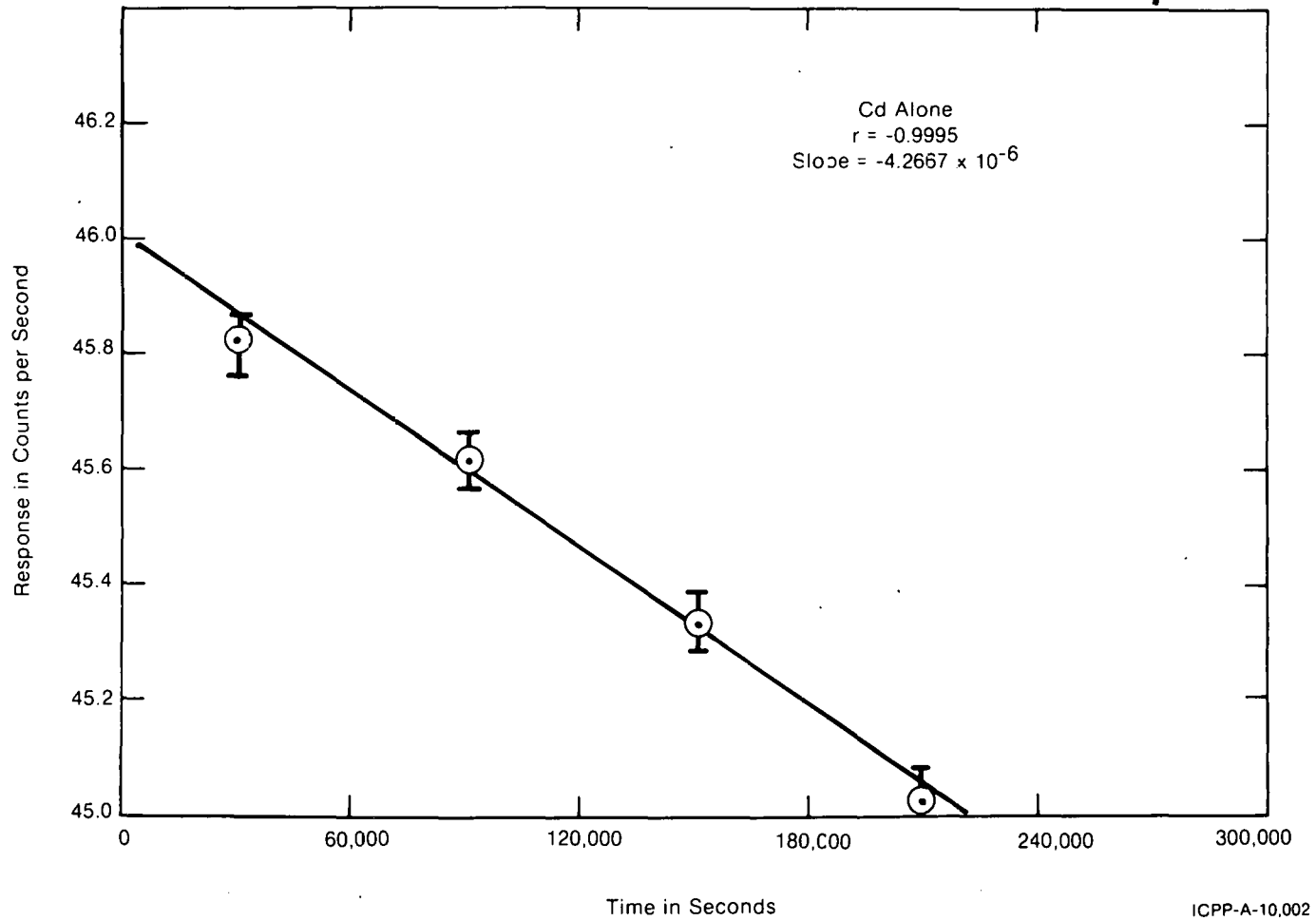
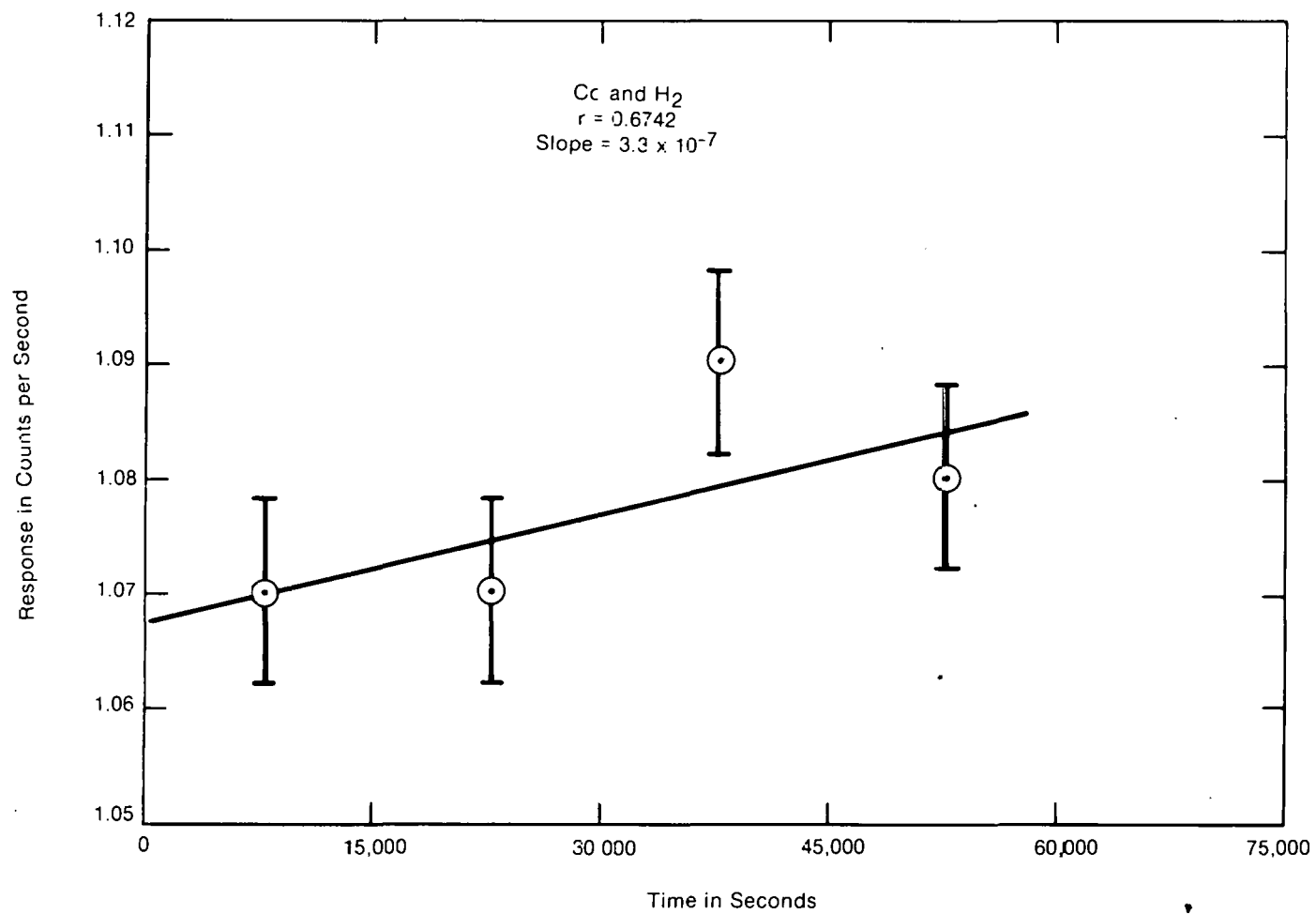


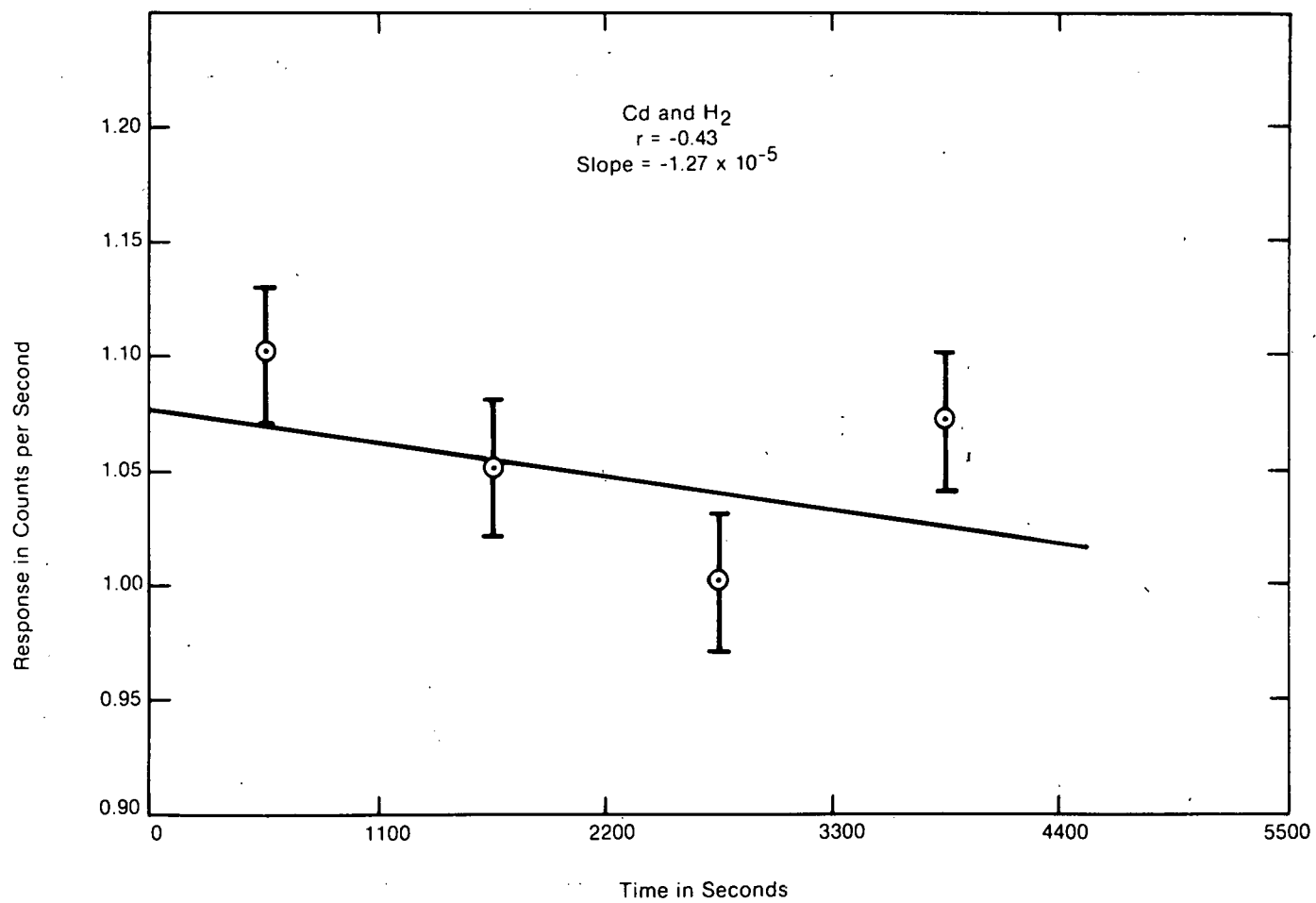
Figure A-10. Response vs Time - Test 10



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Figure A-11. Response vs Time - Test 11

Figure A-12. Response vs Time - Test 12



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Figure A-13. Response vs Time - Test 13

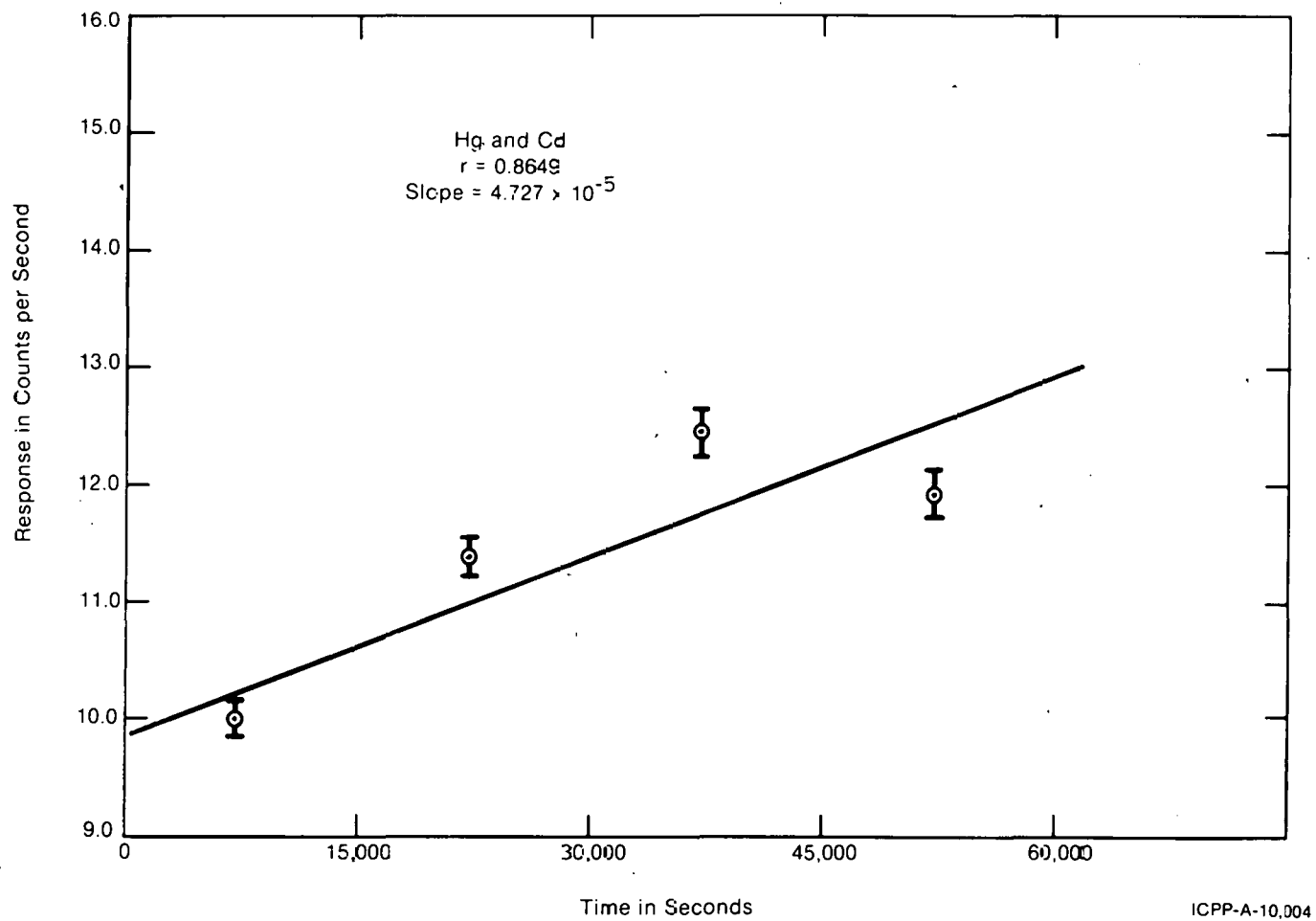
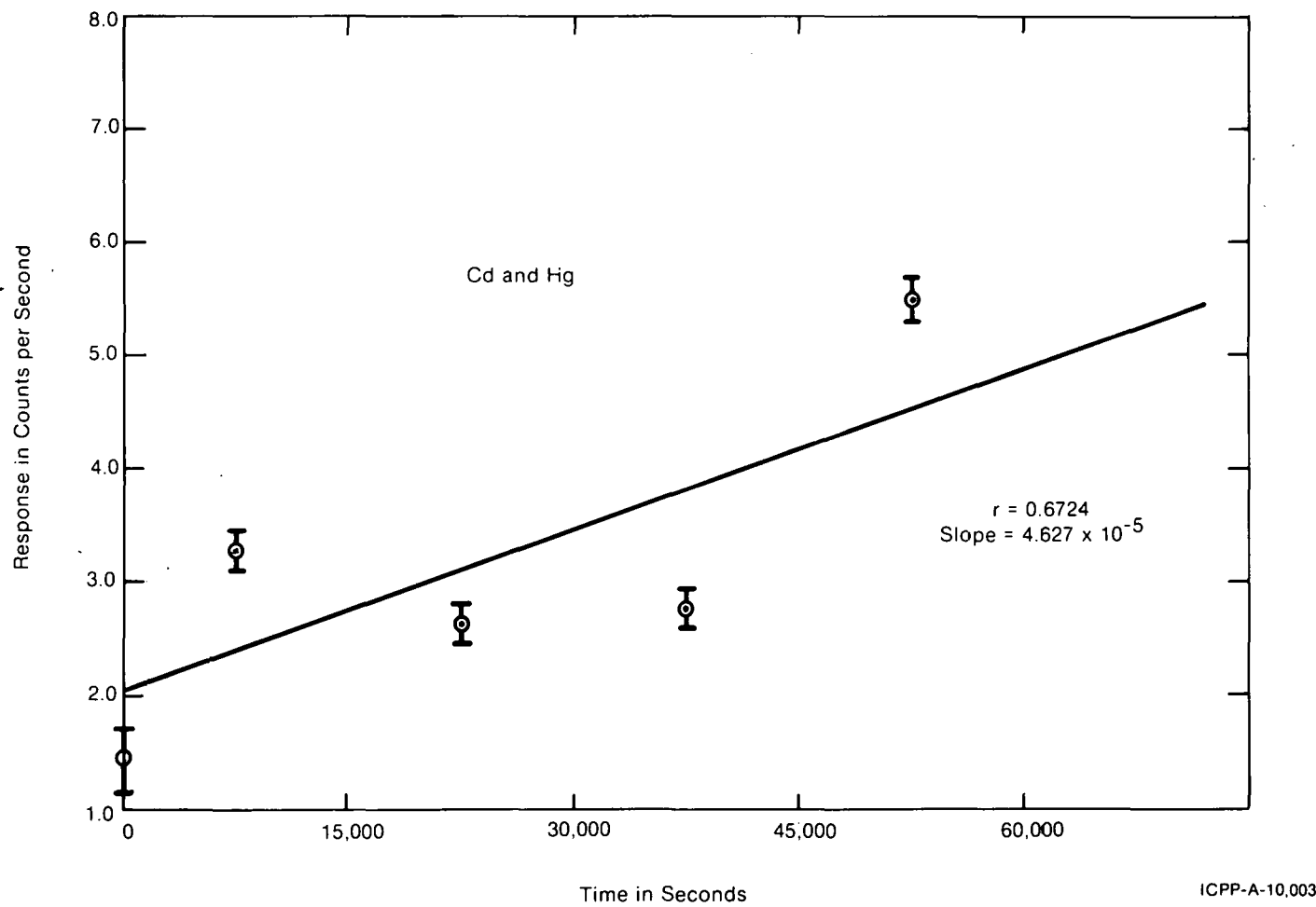


Figure A 14. Response vs Time - Test 14



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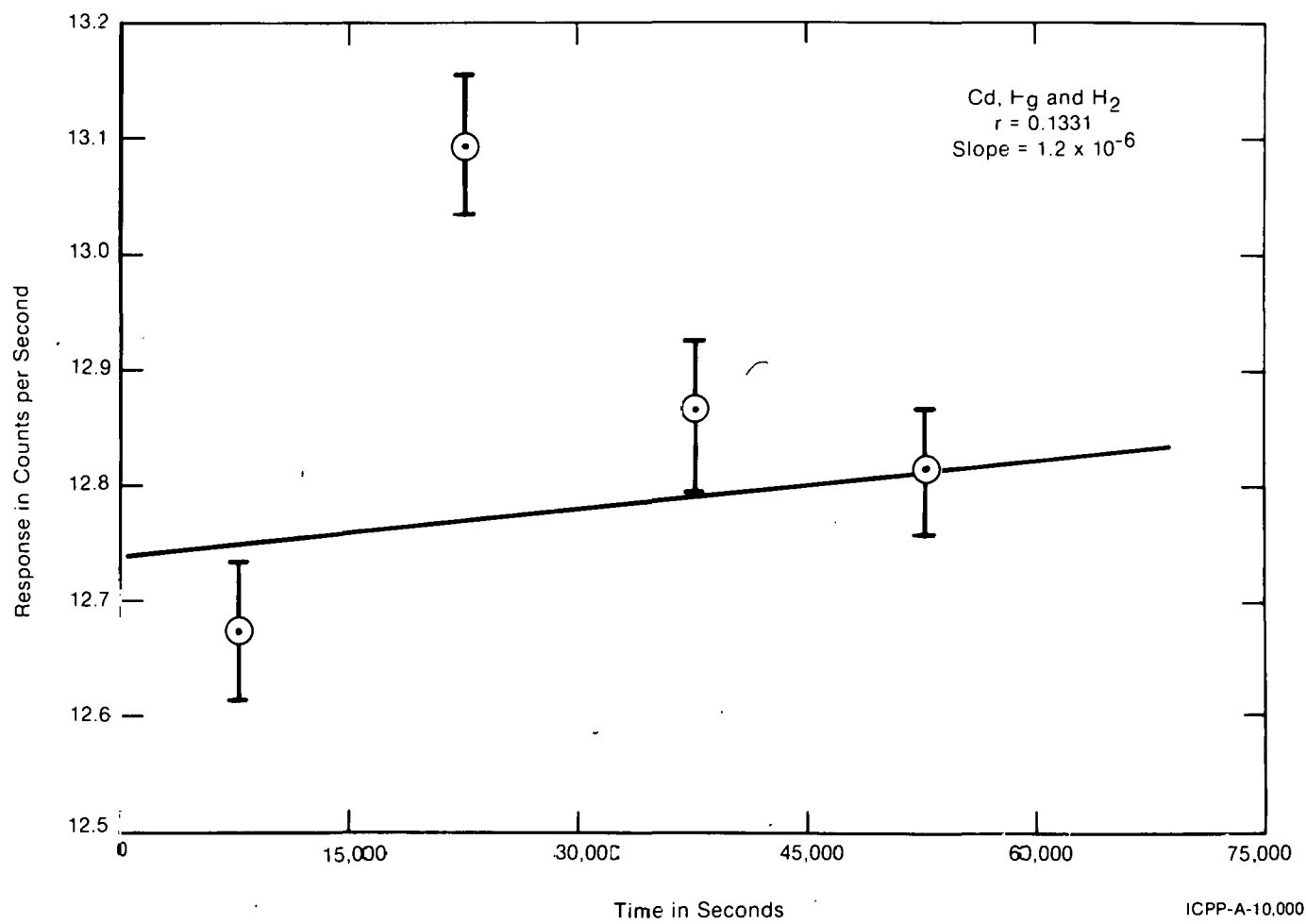
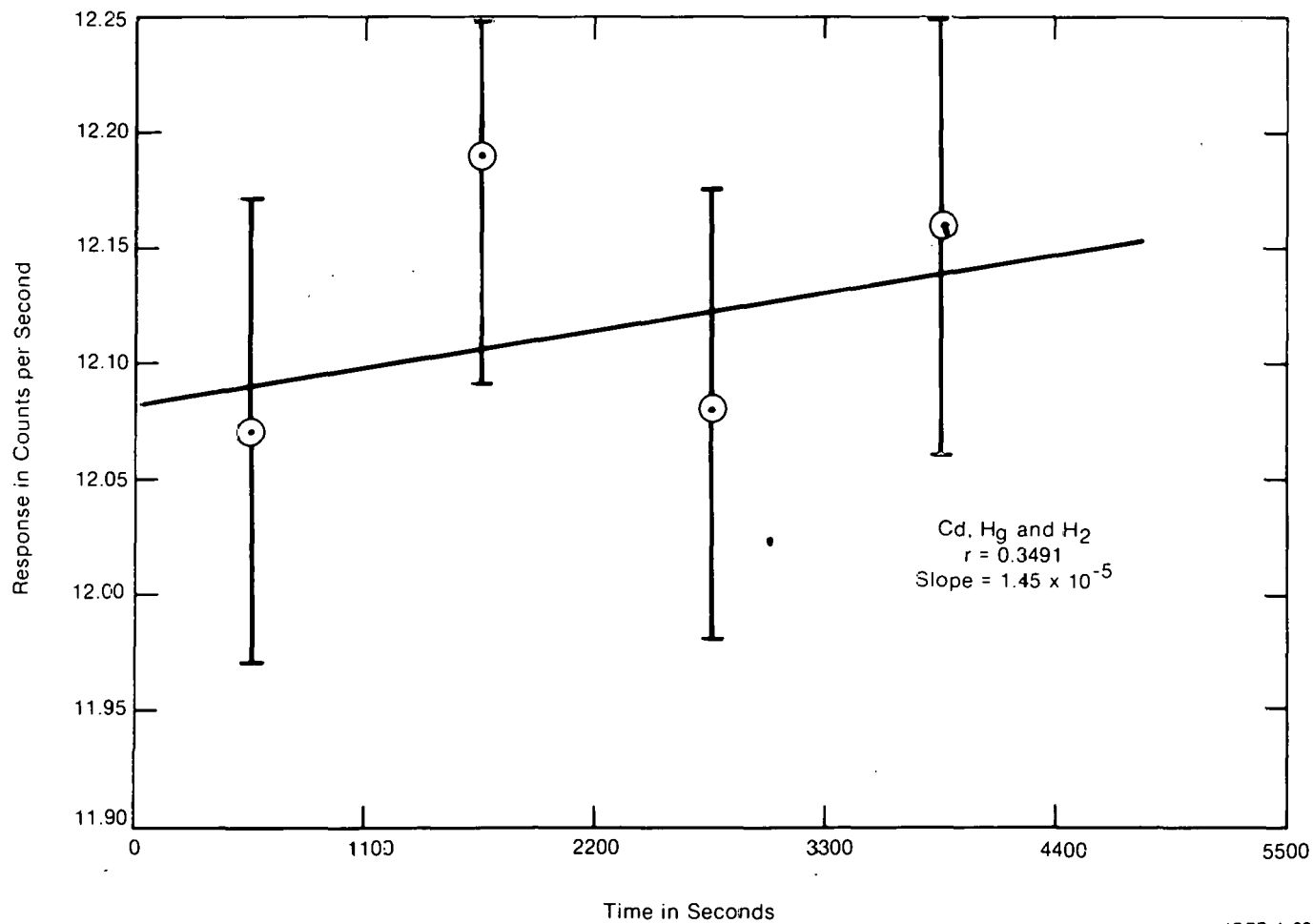


Figure A-15. Response vs Time. - Test 15



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Figure A-16. Response vs Time - Test 16

APPENDIX II

The following Tables present the counting data from the 16 runs.

TABLE A-I
COUNTING DATA - TEST 1

<u>Elapsed Time</u> <u>(sec)</u>	<u>Countrate</u> <u>(cps)</u>
550	2.57
1650	2.77
2750	3.06
3850	3.25

TABLE A-II
COUNTING DATA - TEST 2

<u>Elapsed Time</u> <u>(sec)</u>	<u>Countrate</u> <u>(cps)</u>
550	10.83
1650	10.69
2750	10.83
3850	11.41
4950	12.22
6050	12.29
7150	12.54
8250	12.56

TABLE A-III
COUNTING DATA - TEST 3

<u>Elapsed Time</u> <u>(sec)</u>	<u>Countrate</u> <u>(cps)</u>
550	5.25
1650	5.42
2750	5.56
3850	5.69
4950	5.83

TABLE A-IV

COUNTING DATA - TEST 4

<u>Elapsed Time</u> <u>(sec)</u>	<u>Countrate</u> <u>(cps)</u>
550	6.09
1650	6.41
2750	6.69
3850	7.54
4950	7.60
6050	7.56
7150	7.87

TABLE A-V

COUNTING DATA - TEST 5

<u>Elapsed Time</u> <u>(sec)</u>	<u>Countrate</u> <u>(cps)</u>
0	0.92
30,000	1.05
90,000	1.36
150,000	1.42
210,000	1.43

TABLE A-VI

COUNTING DATA - TEST 6

<u>Elapsed Time</u> <u>(sec)</u>	<u>Countrate</u> <u>(cps)</u>
0	16.45
550	16.62
1650	16.80
2750	16.68
3850	16.70
4950	16.83
6050	16.75
7150	16.81
8250	16.99

TABLE A-VII
COUNTING DATA - TEST 7

<u>Elapsed Time</u> <u>(sec)</u>	<u>Countrate</u> <u>(cps)</u>
1650	16.25
2750	16.28
3850	16.26
4950	16.47
6050	16.69
7150	16.62

TABLE A-VIII
COUNTING DATA - TEST 8

<u>Elapsed Time</u> <u>(sec)</u>	<u>Countrate</u> <u>(cps)</u>
6250	12.65
18750	14.55
31250	14.98
43750	15.68

TABLE A-IX
COUNTING DATA - TEST 9

<u>Elapsed Time</u> <u>(sec)</u>	<u>Countrate</u> <u>(cps)</u>
0	1.00
10,000	1.03
30,000	1.03
50,000	1.04
70,000	1.05

TABLE A-X

COUNTING DATA - TEST 10

<u>Elapsed Time</u> <u>(sec)</u>	<u>Countrate</u> <u>(cps)</u>
30,000	45.85
90,000	45.61
150,000	45.33
210,000	45.09

TABLE A-XI

COUNTING DATA - TEST 11

<u>Elapsed Time</u> <u>(sec)</u>	<u>Countrate</u> <u>(cps)</u>
7500	1.07
22500	1.07
37500	1.09
52500	1.08

TABLE A-XII

COUNTING DATA - TEST 12

<u>Elapsed Time</u> <u>(sec)</u>	<u>Countrate</u> <u>(cps)</u>
550	1.10
1650	1.05
2750	1.00
3850	1.07

TABLE A-XIII

COUNTING DATA - TEST 13

<u>Elapsed Time</u> <u>(sec)</u>	<u>Countrate</u> <u>(cps)</u>
7500	9.98
22500	11.31
37500	12.40
52500	11.98

TABLE A-XIV
COUNTING DATA - TEST 14

<u>Elapsed Time</u> <u>(sec)</u>	<u>Countrate</u> <u>(cps)</u>
0	1.43
7500	3.24
22500	2.68
37500	2.78
52500	5.52

TABLE A-XV
COUNTING DATA - TEST 15

<u>Elapsed Time</u> <u>(sec)</u>	<u>Countrate</u> <u>(cps)</u>
7500	12.67
22500	13.09
37500	12.85
52500	12.81

TABLE A-XVI
COUNTING DATA - TEST 16

<u>Elapsed Time</u> <u>(sec)</u>	<u>Countrate</u> <u>(cps)</u>
550	12.07
1650	12.19
2750	12.08
3850	12.16

APPENDIX III

This appendix presents a sample calculation of the effects and interactions of a 2^3 factorial design. To calculate the H_2 effect from Table II:

$$H_2 \text{ effect} = \frac{(\sum \text{response of runs with } H_2 - \sum \text{response of runs without } H_2)}{(\text{number of runs})/2}$$

From Table II

$$\sum \text{response of runs with } H_2 = 51 + 110 + 34 + 30 + 0.13 - 5.0 + 0.48 + 5.7 = 226.31$$

$$\sum \text{response of runs without } H_2 = 91 + 109 + 1.1 + 17 + 0.23 - 1.7 + 18.7 + 18.3 = 258.63$$

$$H_2 \text{ effect} = \frac{226.31 - 258.63}{16/2} = -3.42\%$$

To calculate the $H_2 + H_g$ interaction

$H_g + H_2$ interaction =

$$\frac{1}{8} \left(\begin{aligned} &\sum \text{response of runs containing both } H_g \text{ and } H_2 + \\ &\sum \text{response of runs containing neither } H_g \text{ and } H_2 - \\ &\sum \text{response of runs containing either } H_g \text{ or } H_2 \text{ but not both} \end{aligned} \right)$$

From Table II

$$\sum (\text{response of runs containing both } H_g \text{ and } H_2) = 34 + 30 + 0.48 + 5.7 = 70.18$$

$$\sum (\text{response of runs containing neither } H_g \text{ or } H_2) = 91 + 109 + 0.23 - 1.7 = 198.53$$

$$\sum (\text{response of runs containing either } H_g \text{ or } H_2 \text{ but not both}) = 51 + 110 + 1.1 + 17 + 0.13 - 5.0 + 18.7 + 18.3 = 211.23$$

$$H_2 + H_g \text{ interaction} = \frac{70.18 + 198.53 - 211.23}{8} = + 7.19\%$$