The use of sodium formate for the recovery of precious metals from acidic base metal effluents

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Abstract: Zinc was used for the reduction of the platinum group metals (PGMs) in acidic effluents. Due to the increasing cost of zinc, sodium formate was investigated as an alternative reductant. In a base metal-containing acidic effluent, called diethylenetriamine barren, sodium formate was used to precipitate the PGMs. This effluent was the filtrate obtained after a precipitation procedure had been used to remove rhodium and iridium. It was found that pH 1.5 was the optimum starting pH for sodium formate reduction. The pH increased to approximately 4.5 after the addition of sodium formate. The optimum concentration of sodium formate was found to be 30 g dm⁻³ at a temperature of 100 °C where the process time was 6h. Platinum and palladium were the most effectively reduced PGMs, both exhibiting an average precipitation efficiency of greater than 99%. Difficulty was experienced with the precipitation of iridium (average precipitation efficiency of 76%). The precipitated PGMs readily dissolved in hydrochloric acid (6M) and sodium chlorate (2%). A reduction in costs resulted from the discontinuation of the use of zinc for reduction purposes. An additional advantage was that zinc was no longer introduced into the PGM refinery circuits.

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INTRODUCTION

The diethylenetriamine barren was an acidic effluent containing base metals (average concentrations: copper $8936\,\mu g\,cm^{-3}$, nickel $1602\,\mu g\,cm^{-3}$, iron $12\,207\,\mu g\,cm^{-3}$ and lead $2511\,\mu g\,cm^{-3}$) and was an industrial hydrochloric acid effluent from Western Platinum Refinery. Full composition details of this effluent are shown in Table 1. Recovery of platinum group metals (PGMs) from this effluent was previously accomplished by using zinc. The average concentrations of PGMs in diethylenetriamine barren were platinum $1026\,\mu g\,cm^{-3}$, palladium $292\,\mu g\,cm^{-3}$, iridium $224\,\mu g\,cm^{-3}$, rhodium $201\,\mu g\,cm^{-3}$ and ruthenium $25\,\mu g\,cm^{-3}$.

Due to the increasing cost of zinc and to the coprecipitation of zinc with the reduced PGM precipitate, an alternative reductant was needed. Formic acid or sodium formate heated to 250 °C at a pressure of 400 kPa has been reported for the reduction of heavy metal ions. The method was used in the presence of sulfide or thiocyanate for the precipitation of heavy metals sulfides. Formic acid or sodium formate was used for the reduction of concentrated PGMs after solvent extraction and from precipitated PGM ammonium salts. Sodium formate was therefore chosen to replace zinc in the reduction of PGMs as it had advantages of availability and competitive price.

METHOD

Parameters that were investigated included pH, sodium formate concentration, temperature and time. Thereafter, precipitate flocculation, filtration and dissolution tests were performed. Initial laboratory trials were performed in glass beakers with effluent aliquots of between 0.4 and 1 dm³ which were heated and stirred by means of electrically operated magnetic stirrer hotplates. Chemically pure reagents were used throughout the experiments. E-pH diagrams were generated using special software.^{5,6} These diagrams were useful in predicting the behaviour of PGMs at different pH values and reduction potentials. For a specific metal, these diagrams showed the areas where species of different oxidation number occur with respect to pH (x-axis) and reduction potential (yaxis). A standard calomel electrode (SCE) with a platinum foil electrode (Radiometer) was used to determine reduction potentials in experimental solutions. Filtration was performed using Buchner flasks and funnels, or a glass Witts apparatus, connected to a vacuum pump (Neuberger). Plant trials were conducted in 22000 dm³ vessels heated by means of steam injection. Mechanical agitation was provided. The vessels were made of rubber-lined mild steel with conical bottoms fitted with valves.

A rapid colorimetric test for residual PGMs in

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Table 1. Analysis (in µq cm⁻³) of diethylenetriamine barren solution before treatment with sodium formate

		Batch number										
Metal	1	2	3	4	5	6	7	8	9	10	11	Average
Pt	547	1961	1738	1293	783	671	1124	952	1126	633	459	1026
Pd	145	337	332	479	372	172	242	337	285	245	271	292
Au	2	10	4	7	4	3	7	2	5	2	2	4
Rh	117	201	161	190	232	265	265	195	187	208	191	201
Ru	191	>1	>2	14	>2	>1	24	6	>1	>1	35	25
lr	16	234	175	229	266	211	268	209	359	207	292	224
Cu	6118	12900	10961	7501	6581	9848	9330	7103	9249	10910	7791	8936
Ni	1677	2318	1879	1855	1706	1489	1554	1485	1373	947	1340	1602
Fe	15966	13900	11383	9665	10700	12100	13200	11990	9836	11460	14076	12207
Pb	2804	5368	3537	2555	2119	3073	1160	1708	2146	1969	1183	2511
Ag	16	68	55	108	33	24	85	22	38	33	43	48
Al	279	352	221	245	189	150	327	89	298	257	196	237
Se	171	224	101	159	254	168	174	100	147	204	251	178
As	1250	1358	1325	1209	1411	1324	1267	1297	1259	1214	1268	1289
Zn	39	21	45	56	85	81	25	57	55	81	45	54

solution was developed⁹ using potassium iodide and stannous chloride solutions.

pH considerations

For the purpose of studying the effect of pH, a concentration of 30 g dm⁻³ of sodium formate (technical grade added in the crystalline form) was chosen. This was based on previous work, 10 where the reduction of PGMs in alkaline effluents was studied. Initial experiments were thus performed on diethylenetriamine barren using sodium formate at 30 g dm⁻³ at 100 °C. The initial pH of diethylenetriamine barren was approximately pH 0 with a hydrochloric acid concentration of approximately 6 M. The starting pH levels of the effluents were adjusted using sodium hydroxide solution (50%). Experiments were carried out on diethylenetriamine barren solution, using a range of pH (pH -0.7 to pH 2.7), in order to determine the optimum starting pH for the sodium formate process at $30 \,\mathrm{g}\,\mathrm{dm}^{-3}$ and $100\,^{\circ}\mathrm{C}$.

Effect of sodium formate addition

Initial laboratory experiments were performed prior to plant trials. In the laboratory trials, diethylenetriamine barren (0.4 dm³ aliquot) was treated with sodium formate, added in the crystalline form (10–50 g dm⁻³), at 100°C and a starting pH of 1.5. Thereafter, plant trials were performed utilising diethylenetriamine barren, with crystalline sodium formate added (at 30 g dm⁻³, 100°C and starting pH 1.5) to obtain −100 mV (SCE) in the solution. A total of 11 batches of 20000 dm³ were treated in the plant trials.

Effect of temperature

Tests were performed at various temperatures between 20 °C and 100 °C using a starting pH of 1.5 and a sodium formate concentration of 30 g dm⁻³.

Effect of time

After the necessary amount of sodium formate

 $(30\,\mathrm{g\,dm^{-3}})$ was added to the solution being reduced (the reduction potential was below $-100\,\mathrm{mV}$ (SCE) at $100\,^\circ\mathrm{C}$), the temperature and agitation was maintained for periods up to a maximum of 19 h. Samples were taken from these laboratory tests at hourly intervals to determine the effect of time on the reduction of PGMs.

Precipitate flocculation, filtration and dissolution tests

Different flocculants were used to determine the optimum performance of flocculation for the reduced PGMs in diethylenetriamine barren. The filtration rates were determined through three grades of Whatman filter paper, namely, 541, 540 and 542. The degree of dissolution in 6M hydrochloric acid and sodium chlorate (2%) was also determined by weighing the mass of the residue after leaching at 70 °C.

RESULTS AND DISCUSSION pH considerations

The precipitation efficiencies of the PGMs in diethy-

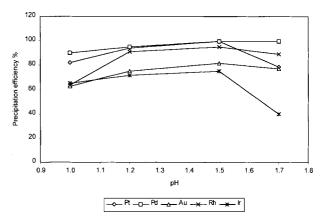


Figure 1. The effect of starting pH on the reduction of PGMs in diethylenetriamine barren using sodium formate (30 g dm⁻³) at 100 °C.

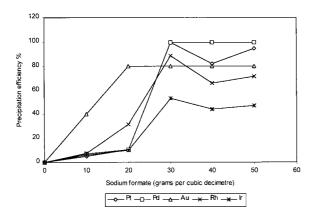


Figure 2. The effect of sodium formate concentration on the reduction of PGMs from diethylenetriamine barren (laboratory trials).

lenetriamine barren increased as the starting pH increased to pH 1.5 (Fig 1). Thereafter the precipitation efficiencies for platinum and rhodium decreased. Ruthenium was omitted from Figs 1 and 2 due to the low initial concentrations of this metal in the diethylenetriamine barren.

In a previous study,¹⁰ the PGMs, in reducing alkaline medium, showed the trend of increasing precipitation efficiencies with decreasing pH. In acidic medium, this trend was different. A peak of PGM precipitation efficiency was found at a starting pH of approximately 1.5 with a sharp decrease as the pH decreased below pH 0. Selective photocatalytic reductions of PGMs were also successfully performed at pH 3.1.¹¹ This fell within the range of pH (1.5 –4.0) of the sodium formate reduction of PGMs.

Effect of sodium formate addition

The reduction of PGMs in diethylenetriamine barren using sodium formate

In the laboratory trials, palladium exhibited excellent precipitation efficiency (98% at 30 g dm⁻³ sodium formate concentration), see Fig 2. The reason for this was thought to be the enhancement of sodium formate reduction properties by finely divided palladium. ^{12,13}

Platinum and rhodium both showed the greatest precipitation efficiencies at 30 g dm⁻³ sodium formate concentration (90% and 78% respectively).

In the laboratory trials, the optimum sodium formate concentration appeared to be approximately $30\,\mathrm{g}\,\mathrm{dm}^{-3}$. There was no reason to exceed the sodium formate concentration of $30\,\mathrm{g}\,\mathrm{dm}^{-3}$ since the PGM precipitation efficiencies decreased with sodium formate concentrations greater than $30\,\mathrm{g}\,\mathrm{dm}^{-3}$ sodium formate. The results obtained were in accordance with the expectations from the *E*–pH diagrams where potentials of less than $-100\,\mathrm{mV}$ at pH between 1.5 and 4.0 indicated oxidation states of zero for the PGMs.

Plant trials using sodium formate for the reduction of PGMs in diethylenetriamine barren
Initial plant trials (20000 dm³) confirmed the trends

found in the laboratory. Palladium exhibited consistently good precipitation efficiencies of greater than 99% at 30 g dm⁻³ sodium formate. ¹³ An encouraging 99% precipitation efficiency was also obtained for platinum. Over a period of 1 month, 11 batches of diethylenetriamine barren (20000 dm³ each) were processed in the plant using between 15 and 30 g dm⁻³ sodium formate at 100°C and a starting pH of 1.5. Platinum and palladium precipitation efficiencies, using sodium formate, were very high (averages of greater than 99%). This was particularly encouraging for diethylenetriamine barren because its primary constituent was platinum and palladium. The average precipitation efficiencies for rhodium and iridium were 97% and 76% respectively. Gold and ruthenium average precipitation efficiencies were 53% and 46% respectively but were considered insignificant due to the low initial concentrations of these metals in the original effluent. Silver and gold were also reduced at average precipitation efficiencies of 85% and 54%, thus contributing to the commercial value of precipitate. Also advantageous was the low precipitation efficiencies for copper, nickel, iron, lead, aluminium and zinc (53%, 25%, 25%, 34%, 29%, and 46% respectively) which resulted in precipitates having relatively low concentrations of these metals. This improved the grade of the PGMs in the precipitates. On the other hand, the high average levels of arsenic and selenium in the precipitates (55% and 88% respectively) resulted in the need for their removal either by means of calcination or hydrometallurgical methods.

Comparison of laboratory and plant trials for diethylenetriamine barren

An interesting feature of the comparison of the laboratory and plant trials for diethylenetriamine barren was the increase in precipitation efficiencies, in the plant trials, for iron, lead, rhodium, and iridium (see Tables 1-4). Opposite trends (decrease in precipitation efficiencies in the plant trials) were noted for copper, nickel, silver, aluminium, selenium, arsenic and zinc. This was beneficial for the concentration of PGMs because the decreased concentration of base metals would effectively increase the concentration of the PGMs. This was inexplicable. The apparent optimum concentration of sodium formate (30g dm⁻³) was the same as that found in the previous study, 10 where 30 g dm⁻³ sodium formate (with sodium thiosulfate at 30 g dm⁻³) was used to recover PGMs from alkaline effluents. Arsenic and selenium in diethylenetriamine barren were reduced by sodium formate. The presence of arsenic and selenium in the precipitate was not considered a problem since they could be removed by calcining or by distillation from the hydrochloric acid medium using cuprous chloride.¹⁴

A disadvantage of calcining was that passivation of rhodium, ruthenium and iridium sometimes occurred. ¹⁵ Diethylenetriamine barren was treated with sodium formate (30 g dm⁻³) on a laboratory scale. At

Table 2. The effect of sodium formate concentration (at 100 °C and pH 1.5) on the reduction of metals from diethylenetriamine barren solution reported as percentage precipitation efficiency based on analyses of barren solutions

	Sodium formate (g dm ⁻³)									
Metal	10	20	30	50	80					
Pt	4.80	10.40	99.60	81.73	94.53					
Pd	7.02	10.79	99.66	99.49	99.49					
Au	40.00	80.00	80.00	80.00	80.00					
Rh	7.58	31.82	88.38	65.66	71.21					
Ru	0.00	0.00	0.00	0.00	0.00					
lr	6.22	10.22	53.33	44.00	47.11					
Cu	5.87	6.52	55.50	43.68	81.56					
Ni	4.15	3.30	41.04	6.91	33.88					
Fe	1.72	7.12	19.83	20.16	11.30					
Pb	4.17	3.53	4.33	5.61	4.65					
Ag	24.48	25.87	99.30	99.30	99.30					
Αl	3.54	8.41	80.09	48.97	32.89					
Se	18.37	72.45	96.94	95.92	93.88					
As	26.79	42.86	64.29	78.57	69.64					
Zn	2.94	0.98	66.67	10.78	22.55					

starting pH 1.5, the maximum precipitation efficiencies were 98%, 98%, and 82% for, palladium, platinum and rhodium respectively.

Copper precipitation increased with increasing sodium formate concentration, having a maximum precipitation efficiency of 81% at 80 g dm⁻³ sodium formate. Nickel precipitation was maximum (41%) at 30 g dm⁻³ sodium formate concentration. Lead precipitated to a small (5% at 30 g dm⁻³ sodium formate concentration) extent. This was not problematic since these concentrations of base metals could be tolerated in the refinery circuits. Silver showed a maximum (99%) of precipitation efficiency at 30 g dm⁻³ sodium formate concentration. This was desirable since silver increased the value of the precipitate.

Effect of temperature

It was evident that optimum temperature for the sodium formate reduction of PGMs in diethylenetriamine barren was reached at 100 °C (Fig 3). Palladium could be precipitated to 98% precipitation efficiency at 70 °C. This was anticipated due to palladium's enhancing effect on the reducing power of sodium formate. ^{12,13} The other PGMs are only precipitated to higher (between 47% and 99%) precipitation efficiencies at 100 °C. This was corroborated by the findings in previous work. ¹⁰ Ruthenium was included in Fig 3 to demonstrate its precipitation characteristics even though it was present at low initial concentrations in the untreated effluent.

Effect of time

Palladium was efficiently precipitated by sodium formate after 1h. Again, this was thought to be due to the catalytic effect that finely divided palladium has on the reducing effect of sodium formate. For iridum, the percentage metal remaining in solution becomes almost constant, with respect to time, at approximately 25% of the original concentration. The reason for this was thought to be the existence of a very stable iridium complex that is not reduced by very low (-100 mV SCE at 100 °C) reduction potentials. A processing time of 6h was required to produce maximum precipitation of the PGMs.

Precipitate flocculation, filtration and dissolution tests

Flocculation

The anionic and neutral flocculants¹⁶ (acrylamide–acrylic acid co-polymers and poly acrylamides) that were tested, produced precipitates that settled quickly (approximately 30 min for diethylenetriamine barren treated with 0.032% flocculant). The cheapest flocculant (Magnafloc 351 –a non-ionic polyacrylamide) was selected and the following results were obtained. Diethylenetriamine barren treated with sodium for-

Table 3. Analysis (in µg cm⁻³) of diethylenetriamine barren solution after treatment with sodium formate

	Batch number											
Metal	1	2	3	4	5	6	7	8	9	10	11	Average
Pt	>1	8	>1	2	12	3	2	4	>1	>1	3	3
Pd	>1	2	>1	>1	>1	>1	>1	>1	3	>1	2	1
Au	>1	>1	>1	>1	3	>1	>1	>1	2	2	2	1
Rh	>1	3	>1	>1	15	8	11	8	4	5	8	6
Ru	>1	>1	>1	9	>1	>1	>1	>1	>1	>1	>1	2
lr	15	27	67	43	50	36	48	27	33	27	35	37
Cu	6102	7675	7248	2708	2578	6569	1200	3007	4441	4146	>1	4152
Ni	1632	1899	1394	691	1242	1165	1263	1016	965	733	1128	1193
Fe	12300	10500	9844	3549	7280	8596	11500	7739	8723	9822	11300	9196
Pb	1472	4755	2998	8	1790	2513	536	1201	1432	1346	1018	1734
Ag	16	2	18	14	>1	>1	>1	>1	>1	>1	>1	5
Al	196	152	185	168	120	101	259	80	206	221	128	165
Se	7	5	11	12	54	12	19	7	56	21	11	20
As	652	549	421	467	751	611	510	719	439	429	800	577
Zn	15	11	18	20	66	54	10	29	41	54	22	31

Table 4. Percentage precipitation efficiencies of metals reduced from diethylenetriamine barren solutions using sodium formate

	Batch number											
Metal	1	2	3	4	5	6	7	8	9	10	11	Average
Pt	99.82	99.59	99.94	99.85	98.47	99.55	99.82	99.58	99.91	99.84	99.35	99.61
Pd	99.31	99.41	99.70	99.79	99.73	99.42	99.59	99.70	98.95	99.59	99.26	99.50
Au	50.00	90.00	75.00	85.71	25.00	66.67	85.71	50.00	60.00	0.00	0.00	53.46
Rh	99.15	98.51	99.38	99.47	93.53	96.98	95.85	95.90	97.86	97.60	95.81	97.28
Ru	99.48	0.00	50.00	35.71	50.00	0.00	95.83	83.33	0.00	0.00	97.14	46.50
lr	6.25	88.46	61.71	81.22	81.20	82.94	82.09	87.08	90.81	86.96	88.01	76.07
Cu	0.26	40.50	33.87	63.90	60.83	33.30	87.14	57.67	51.98	62.00	99.99	53.77
Ni	2.68	18.08	25.81	62.75	27.20	21.76	18.73	31.58	29.72	22.60	15.82	25.16
Fe	22.96	24.46	13.52	63.28	31.96	28.96	12.88	35.45	11.32	14.29	19.72	25.35
Pb	47.50	11.42	15.24	99.69	15.53	18.22	53.79	29.68	33.27	31.64	13.95	33.63
Ag	0.00	97.06	67.27	87.04	96.97	95.83	98.82	95.45	97.37	96.97	97.67	84.59
Αl	29.75	56.82	16.29	31.43	36.51	32.67	20.80	10.11	30.87	14.01	34.69	28.54
Se	95.91	97.77	89.11	92.45	78.74	92.86	89.08	93.00	61.90	89.71	95.62	88.74
As	47.84	59.57	68.23	61.37	46.78	53.85	59.75	44.56	65.13	64.66	36.91	55.33
Zn	61.54	47.62	60.00	64.29	22.35	33.33	60.00	49.12	25.45	33.33	51.11	46.20

mate after 0.032% flocculant addition produced acceptable precipitation rates.¹⁷ The unflocculated solution did not settle appreciably in the required time of 60 min.

Filtration

Filtration of the precipitates was easily performed through Whatman filter paper grade 540. Similarly, filtration on the plant-scale was carried out without any difficulty. This could be attributed to the absence of any gelatinous type material in the precipitate. The filtration rate was approximately 0.12 dm³ per min on a 150 mm diameter Buchner funnel.

Dissolution of precipitates

All the precipitates from the reduction of PGMs in diethylenetriamine barrens were readily soluble in 6 M hydrochloric acid and sodium chlorate (2%) at 70 °C, provided that the concentration of damp solids (approximately 20% moisture) was not more than 330 g dm⁻³ in the acid solution.

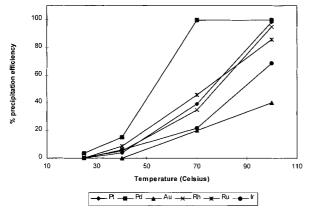


Figure 3. The effect of temperature on the reduction of PGMs in diethylenetriamine barren solution treated with sodium formate.

CONCLUSIONS

Sodium formate was an effective reducing agent for the recovery of PGMs from the acidic base metal effluent, diethylenetriamine barren. This was most advantageous since platinum, palladium and rhodium were the major PGM components of the effluent. Sodium formate was not as effective for the reduction of iridium, however, where it was thought that a stable complex of iridium prevented the complete reduction of this metal. The residual iridium was subsequently removed by an electrolytic precipitation method. A starting pH of 1.5 with sodium formate concentration of 30 g dm⁻³ processed at 100 °C proved to be the most effective in terms of PGM reduction. Under these conditions, the PGMs were precipitated to a maximum in a processing time of 6 h.

The replacement of zinc by sodium formate, as a reductant for PGMs, resulted in a cost saving of approximately 53%. It also removed the PGM precipitate contamination factor associated with using a metal reductant. The use of sodium formate was extremely cost effective (profit to cost ratio of 157) in recovering PGMs from this base metal effluent.

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