

## Performance characteristics of chloro-substituted dinitrobenzene for magnesium reserve batteries

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### Abstract

The electrochemical characteristics of 1-chloro-2,4-dinitrobenzene (CDNB) as a cathode depolarizer in a cell with a magnesium anode is investigated at different temperatures (+28 to  $-10$  °C). The discharge behaviour of  $\text{Mg}/\text{Mg}(\text{ClO}_4)_2/\text{CDNB}$  cells shows high cathode potential and efficiency. Resonance effects contribute to the higher degree of electroreduction of the nitro groups in the substituted compounds and this results in the higher efficiency. Cyclic voltammetric (CV) studies of CDNB in neutral battery electrolyte at a glassy-carbon electrode substantiate the observed reduction behaviour.

### Introduction

A suitable energy-storage system is an essential requirement for remote power supplies that use photovoltaic arrays. For such applications, low cost and simplicity of operation are major requirements and, therefore, dry cells are employed. The latter, however, have a limited shelf life. This difficulty can be overcome by the use of reserve batteries [1]. In particular, magnesium reserve systems have recently received considerable attention as they promise to be inexpensive and possess many attractive features such as long shelf life, high energy density, flexible design, and excellent performance at sub-zero temperatures [1].

For a number of years, the authors have been involved in the development of high-energy magnesium/organic systems. In the case of magnesium/inorganic systems, inorganic depolarizers such as  $\text{MnO}_2$ ,  $\text{CuO}$ ,  $\text{HgO}$ , etc., have inherent limited coulombic capacity output in comparison with organic depolarizers. This is because inorganic materials undergo one- or two-electron transfer, whereas organic compounds are capable of 6- to 12-electron transfer, e.g., nitro and nitroso compounds [2–10].

The substitution effect in the benzene nucleus on the performance of dinitro compounds has been the subject of various studies [2, 3, 11]. It has been found that electron-withdrawing substituents will enhance the operating potential of a cell while electron-donating groups will reduce the potential. Based on the 12-electron transfer process, the theoretical coulombic capacity of 1-chloro-2,4-dinitrobenzene (CDNB) is  $1.580 \text{ Ah g}^{-1}$ . The operating voltage of the  $\text{Mg}/\text{Mg}(\text{ClO}_4)_2/\text{CDNB}$  system is higher than that of the  $\text{Mg}/\text{Mg}(\text{ClO}_4)_2/m\text{-DNB}$  counterpart. In this paper, the electrochemical characteristics of CDNB are examined by discharging cells with magnesium perchlorate electrolyte and different catalysts at different current densities and temperatures.

Cyclic voltammetric (CV) studies of CDNB in the above electrolyte have also been conducted.

## Experimental

### *Cell fabrication and discharge study*

Magnesium alloy (Magnesium Elektron Ltd, UK) sheets (AZ 31 consisting of 2.5 to 3.5 wt.% Al, 0.6 to 1.4 wt.% Zn and 0.15 to 0.70 wt.% Mn) of dimensions 4 cm × 6 cm × 0.5 cm served as the anode. A copper grid of the same dimensions as that of magnesium sheets was used as the current collector for the cathodes. The cathode was prepared using 3 g of CDNB (E. Merck), different concentrations (10 to 70 wt.%) of acetylene black, and 2.5 cm<sup>3</sup> of an aqueous solution of 2 to 3% carboxymethylcellulose (BDH) binder. Various additives such as CuO (BDH), Fe<sub>2</sub>O<sub>3</sub> (BDH) and V<sub>2</sub>O<sub>5</sub> (BDH) were also added to the optimized cathode mix during the preparation of the plate. These cathodes were pressed at an optimum pressure (420 kg cm<sup>-2</sup>). In each cell, the cathode was placed between two anodes and separated by cellophane paper. The cells were assembled in a polyvinylchloride (PVC) container and activated with 2 M magnesium perchlorate electrolyte. Sufficient time was allowed for the complete wetting of electrodes before commencing the discharge process. The cells were discharged at a current drain of 200, 300 or 400 mA. All room-temperature (28 ± 1 °C) and low-temperature (0, 10 and -10 ± 1 °C) experiments were performed using a thermostat (Colora thermostat, Germany).

### *Half-cell potential*

In these experiments, a Ag/AgCl reference electrode (3.0 M NaCl) was connected to the organic cathode and the half-cells potentials of cathodes were measured at initial and final stages at a constant temperature of 28 ± 1 °C.

### *Cyclic voltammetric studies*

A BAS 100A Electrochemical Analyser and a low-current Module (Bio Analytical System Inc., West Lafayette, IN, USA) was used for CV studies of the electrochemical behaviour of CDNB. A digital plotter (DMF-40 Series Houston Instrument Division) and a dotmatrix printer (Panasonic KX-Plosoi, NJ, USA) were employed for recording the results.

Experiments were performed with a standard, three-electrode, glass cell with a nitrogen inlet for de-aeration, a bubbler for gas exit, a water inlet and an outlet for temperature maintenance. The studies were carried out at 28 ± 1 °C.

The working electrode was a glassy-carbon electrode of area 0.0707 cm<sup>2</sup>. This was embedded in Teflon. A Ag/AgCl reference electrode and a platinum-foil auxiliary electrode comprised the remainder of the electrochemical cell. The glassy-carbon electrode was polished with alumina polish on microcloth for 2 min and then cleaned with distilled water. This was followed by the ultrasonic cleaning for 5 min, to remove any residual alumina particles from the surface, and, finally, the electrode was rinsed with acetone.

The CV profiles were recorded over a potential range of -300 to -900 mV at various scan rates (viz., 5, 10, 20, 40, 80, 160 and 320 mV s<sup>-1</sup>). The concentration of the CDNB solution was varied from 10 to 30 mM. The reproducibility of the cyclic voltammograms was ± 2% which was verified periodically by recording the response under identical conditions. Purified nitrogen gas was bubbled in the electrolytic cell

before each experiment. The reference electrode was connected through a salt bridge. In analysing the experimental data, care was taken to subtract the background current at each scan rate.

The CDNB depolarizer (Fluka AG, Buchs, Switzerland) was dissolved in 10% ethanol solution and made up with the supporting electrolyte to the desired concentration. The magnesium perchlorate supporting electrolyte (E. Merck, AR, Germany) was prepared with triple-distilled water.

## Results and discussion

### Room-temperature studies

#### Capacity with different concentrations of acetylene black

To optimize the concentration of the conducting material (acetylene black), the cells were fabricated with different concentrations of acetylene black, viz., 0, 10, 20, 30, 40, 50, 60 and 70 wt.% of CDNB. The corresponding cells were discharged at a constant current of 200 mA and the resulting capacities were 0.15, 0.3, 0.5, 1.1, 1.4, 1.6, 1.70 and 1.75 Ah, respectively. Clearly, the increase in capacity due to an increase in acetylene black above 50 wt.% is not significant. Given this finding and considering the high bulk density of acetylene black, the composition corresponding to 50 wt.% acetylene black was taken as the optimum value (Fig. 1). The inset of Fig. 1 illustrates the voltage versus current relationship for different concentrations of acetylene black. The observed linearity at each current drain is indicative of the fact that the polarization

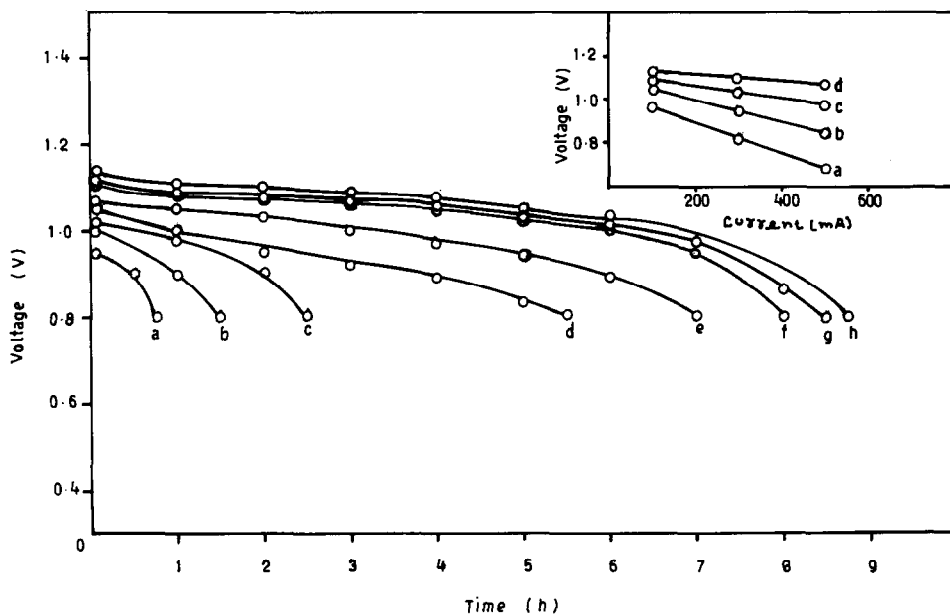


Fig. 1. Effect of acetylene black on Mg/Mg(ClO<sub>4</sub>)<sub>2</sub>/CDNB cells. Current drain: 200 mA, and temperature: 28 ± 1 °C. Concentration of acetylene black: (a) 0%; (b) 10%; (c) 20%; (d) 30%; (e) 40%; (f) 50%; (g) 60%, and (h) 70%. Inset: variation of voltage with current drain for different acetylene black contents (wt.%): (a) 10; (b) 30; (c) 50, and (d) 70.

is ohmic controlled. It is noted that the internal resistance (slope value) for 50 wt.% acetylene black was lower than that for either 30 or 10 wt.%. When compared with higher concentration (viz., 70 wt.%), however, there is little difference in the internal resistance. Hence, a cell with 50 wt.% acetylene black reaches maximum capacity.

#### Discharge characteristics

The half-cell potential of the CDNB cathode was measured against a Ag/AgCl reference electrode at 200 mA current drain and the results are presented in Table 1. The half-cell potential of the CDNB cathode (+0.220 V) and the closed-circuit voltage (CCV) of the CDNB cell are much higher than those using the unsubstituted nitro compound (half-cell potential=0.120 V). This is due to the presence of an electrophilic chlorine atom in the benzene ring. Moreover, the compound exhibits a higher discharge potential (100 mV) than the *m*-dinitrobenzene until the end of the discharge because of the superior discharge capability of the depolarizer.

The greater ampere-hour efficiency of CDNB compared with *m*-DNB is due to the chlorine species in the dinitrobenzene. Usually, the presence of chlorine in the benzene nucleus causes a net increase in electron density at the *ortho*- and *para*-positions due to the predominant resonance effect (+R) rather than an inductive effect (-I) [12]. A similar situation exists in CDNB wherein the electron density increases at the *ortho*- and *para*-positions and, in turn, increases the electron density at the -C-NO<sub>2</sub> groups present at the same positions. An increase in the electron density at the -C-NO<sub>2</sub> groups facilitates the protonation. As the protonation reaction is a prerequisite for easy electroreduction, the slightly faster protonation [13] that occurs in CDNB compared with *m*-DNB results in easier reduction of CDNB and, hence, increases the ampere-hour efficiency. These aspects are confirmed by cyclic voltammetry and discharge studies of Mg/CDNB cells.

Cyclic voltammetric data obtained for CDNB and *m*-DNB show that, under the same experimental conditions, the reduction of CDNB takes place at a less negative potential than that of *m*-DNB [14]. This observation supports the observed easier reduction, as well as the higher ampere-hour efficiency, obtained with CDNB cells. Thus, chlorine substitution in *m*-DNB makes CDNB a better depolarizer than *m*-DNB and yields higher ampere-hour efficiency.

The discharge behaviour of the Mg/*m*-DNB and Mg/CDNB cells at different current drains, viz., 200, 300 and 400 mA are shown in Fig. 2(A) and 2(B), respectively. The specific capacity values are tabulated in Table 2. The data show that the cathode polarization increases with increase in the current density and results in a loss of cell

TABLE 1

Half-cell potentials of cathodes at 28 ± 1 °C

Depolarizer	Potential (mV vs. Ag/AgCl)	
	Initial	Final
<i>m</i> -DNB	+120	-515
CDNB	+220	-500
CDNB + CuO	+270	-475
CDNB + Fe <sub>2</sub> O <sub>3</sub>	+250	-490
CDNB + V <sub>2</sub> O <sub>5</sub>	+230	-500

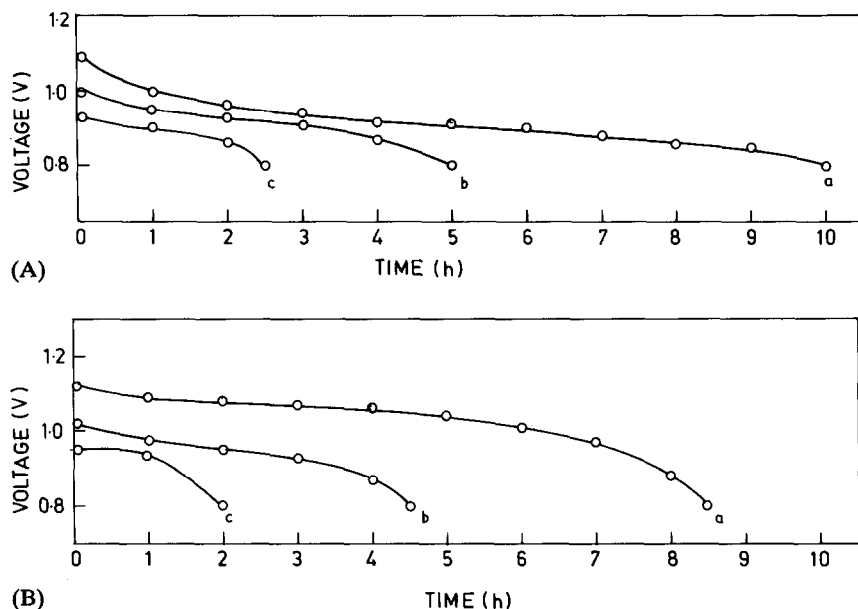


Fig. 2. (A) Effect of current density on  $\text{Mg}/\text{Mg}(\text{ClO}_4)_2/m\text{-DNB}$  cells, and (B) effect of current density on  $\text{Mg}/\text{Mg}(\text{ClO}_4)_2/\text{CDNB}$  cells. Current drain (mA): (a) 200; (b) 300; (c) 400, and temperature:  $28 \pm 1$  °C.

TABLE 2

Cell ampere-hour and watt-hour capacities at different current densities at  $28 \pm 1$  °C

Depolarizer	Capacity ( $\text{Ah g}^{-1}$ )		
	Current density ( $\text{mA cm}^{-2}$ )		
	4.2	6.3	8.3
<i>m</i> -Dinitrobenzene ( <i>m</i> -DNB)	0.66 (0.62)	0.50 (0.45)	0.33 (0.29)
1-Chloro-2,4-dinitrobenzene (CDNB)	0.56 (0.54)	0.45 (0.41)	0.27 (0.24)

Values in parenthesis are the Wh capacities of the cell.

capacity. Figure 3 shows the dependence of the cell capacity on current density or concentration of acetylene black. The cell reactions are as follows.

At anode:



at cathode:



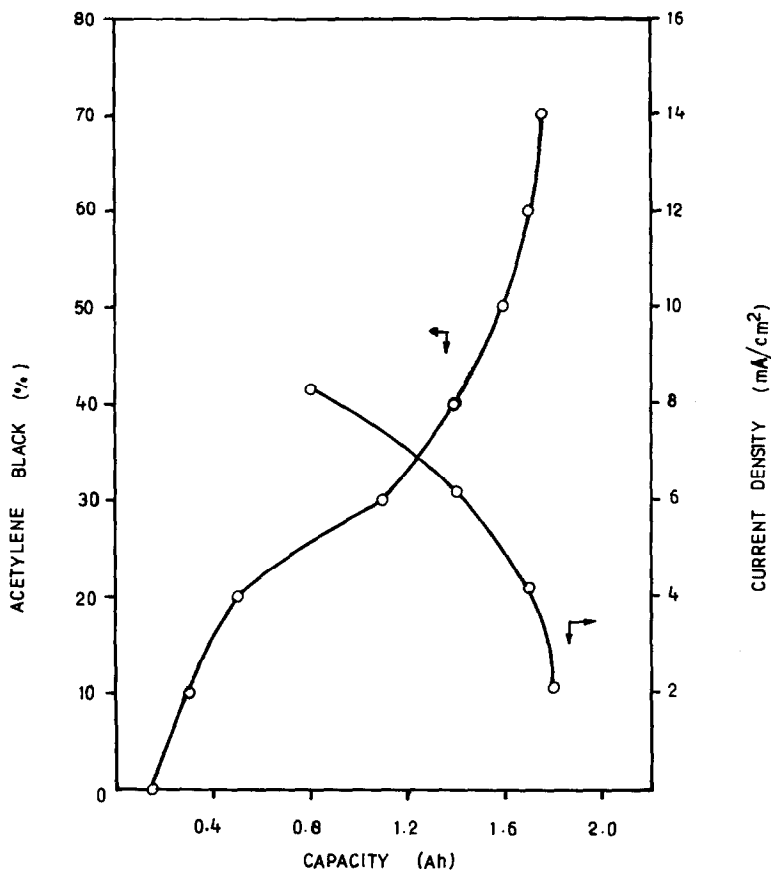


Fig. 3. Acetylene black and current density vs. capacity curves for Mg/Mg(ClO<sub>4</sub>)<sub>2</sub>/CDNB cells.

#### *Effect of catalyst on cell performance*

The reduction efficiency of organic compounds depends on the pH of the electrolyte, the current drain, and the catalyst used. To improve the reduction efficiency of organic compounds, different inorganic catalysts have been examined [15, 16]. In this paper, a study has been made of the effect of different metal oxide catalysts, i.e., CuO, Fe<sub>2</sub>O<sub>3</sub> and V<sub>2</sub>O<sub>5</sub>, on the performance of CDNB in magnesium batteries at different current drains, namely, 200, 300 and 400 mA, see Figs. 4 to 6.

At a 200 mA current drain, the cells gave only 0.56 Ah g<sup>-1</sup> without any catalysts. By contrast, the addition of 2 to 3% (i.e., wt.% of CDNB) CuO, Fe<sub>2</sub>O<sub>3</sub> and V<sub>2</sub>O<sub>5</sub> enhanced the capacity output to 0.66, 0.60 and 0.54 Ah g<sup>-1</sup>, respectively. This performance is due to the catalytic effect of the inorganic compound, a reduction in the internal resistance of the cell during discharge (Fig. 7), and an increase in the hydrogen overvoltage of the organic compound. The Ah g<sup>-1</sup>, Wh g<sup>-1</sup> capacities and cathode efficiency of the cells are presented in Table 3.

The catalysts improve the capacity of the cells at room and low temperature in the order: CuO > Fe<sub>2</sub>O<sub>3</sub> > V<sub>2</sub>O<sub>5</sub>. At room temperature, the increase in capacity caused by CuO and Fe<sub>2</sub>O<sub>3</sub> is 7% and 3%, respectively; in the case of V<sub>2</sub>O<sub>5</sub>, the effect is

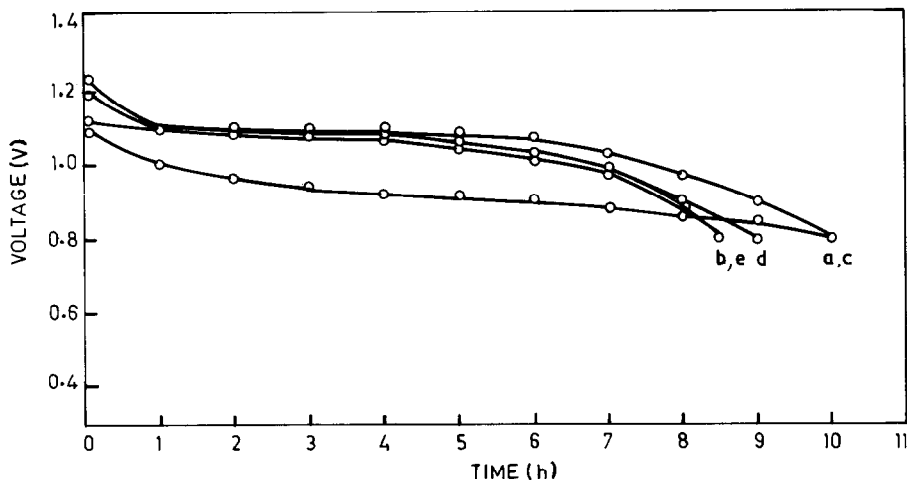


Fig. 4. Effect of catalyst on Mg/Mg(ClO<sub>4</sub>)<sub>2</sub>/CDNB cells. Current drain: 200 mA, and temperature: 28 ± 1 °C. (a) *m*-DNB alone; (b) CDNB alone; (c) CDNB + CuO, (d) CDNB + Fe<sub>2</sub>O<sub>3</sub>, and (e) CDNB + V<sub>2</sub>O<sub>5</sub>.

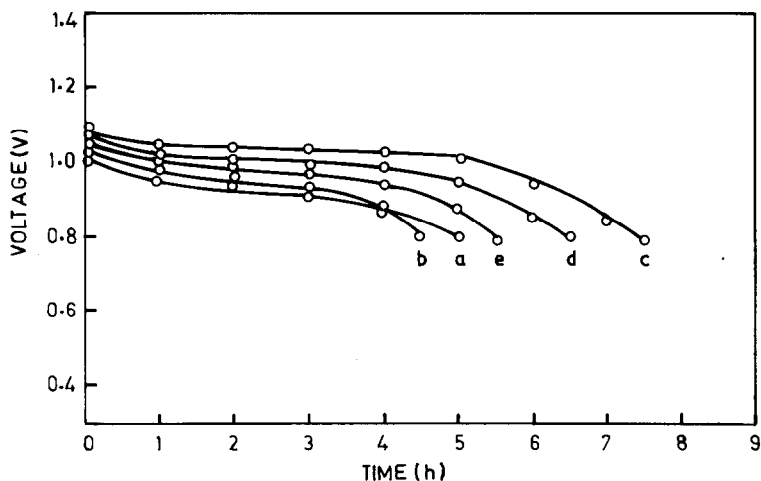


Fig. 5. Effect of catalyst on Mg/Mg(ClO<sub>4</sub>)<sub>2</sub>/CDNB cells. Current drain: 300 mA, and temperature: 28 ± 1 °C. (a) *m*-DNB alone; (b) CDNB alone; (c) CDNB + CuO; (d) CDNB + Fe<sub>2</sub>O<sub>3</sub>, and (e) CDNB + V<sub>2</sub>O<sub>5</sub>.

negligible. At low temperature (+10 °C), the efficiency increases due to CuO and Fe<sub>2</sub>O<sub>3</sub> is 3% and 1%, respectively; again V<sub>2</sub>O<sub>5</sub> produces no effect.

#### Low-temperature studies

To evaluate the capacity of the above cells at low temperatures, the cells were discharged at 10, 0 and -10 °C. The results are presented in Fig. 8. The Ah and Wh capacity values are tabulated in Table 4. As can be seen from Fig. 8, the capacities of the above cells at +10 °C with catalysts CuO, Fe<sub>2</sub>O<sub>3</sub> and V<sub>2</sub>O<sub>5</sub> are 0.20, 0.18 and

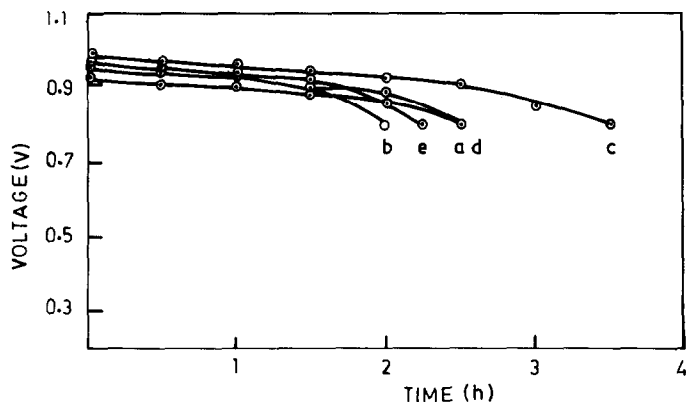


Fig. 6. Effect of catalyst on Mg/Mg(ClO<sub>4</sub>)<sub>2</sub>/CDNB cells. Current drain: 400 mA, and temperature: 28 ± 1 °C. (a) *m*-DNB alone; (b) CDNB alone; (c) CDNB + CuO; (d) CDNB + Fe<sub>2</sub>O<sub>3</sub>, and (e) CDNB + V<sub>2</sub>O<sub>5</sub>.

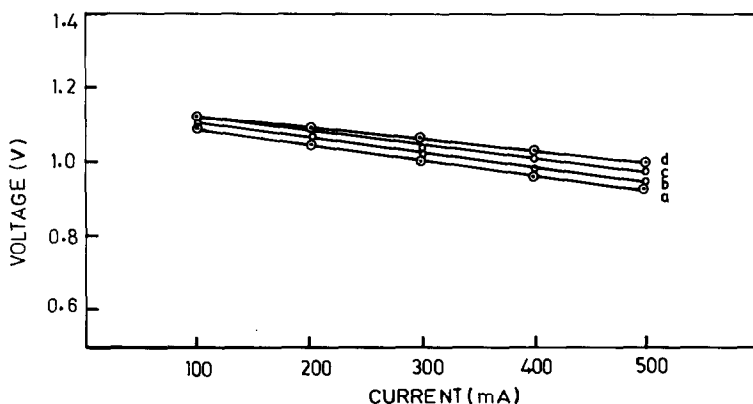


Fig. 7. Variation of voltage with current drain for different catalysts: (a) CDNB alone; (b) CDNB + V<sub>2</sub>O<sub>5</sub>; (c) CDNB + Fe<sub>2</sub>O<sub>3</sub>, and (d) CDNB + CuO.

0.16 Ah g<sup>-1</sup>, respectively. The order of improvement is the same as that found at room temperature. Furthermore, the drop in the discharge voltage is less in the case of CuO and more in the case of V<sub>2</sub>O<sub>5</sub> and Fe<sub>2</sub>O<sub>3</sub>. When the temperature is lowered further to 0 and -10 °C, the capacities of these cells decrease gradually in the same order as that observed at 28 and 10 °C. It is clear, therefore, that the capacity decreases with decrease in temperature as a result of a decrease in conductivity, an increase in the cell internal resistance (see inset of Fig. 8), and a higher electrolyte viscosity [17] at lower temperatures.

#### Cyclic voltammetric studies

Figure 9 shows typical cyclic voltammograms obtained for the reduction of 10 mM CDNB in 2 M magnesium perchlorate solution on a glassy-carbon electrode at different scan rates, viz., 5 to 320 mV s<sup>-1</sup>. Table 5 gives the cyclic peak parameters obtained for the reduction of different concentrations, viz., 10 to 30 mM of CDNB.



TABLE 3

Capacity, energy density and cathode efficiency of cells with different catalysts at  $28 \pm 1$  °C

Depolarizer	Current density ( $\text{mA cm}^{-2}$ )								
	4.2			6.3			8.3		
	A	B	C	A	B	C	A	B	C
<i>m</i> -DNB	0.66	0.62	34	0.50	0.45	26	0.33	0.29	17
CDNB	0.56	0.54	35	0.45	0.41	28	0.27	0.24	17
CDNB + CuO	0.66	0.67	42	0.60	0.56	39	0.47	0.42	30
CDNB + $\text{Fe}_2\text{O}_3$	0.60	0.60	38	0.55	0.51	35	0.33	0.29	21
CDNB + $\text{V}_2\text{O}_5$	0.54	0.52	35	0.50	0.46	32	0.30	0.26	19

A = Capacity ( $\text{Ah g}^{-1}$ ).B = Energy density ( $\text{Wh g}^{-1}$ ).

C = Cathode efficiency (%).

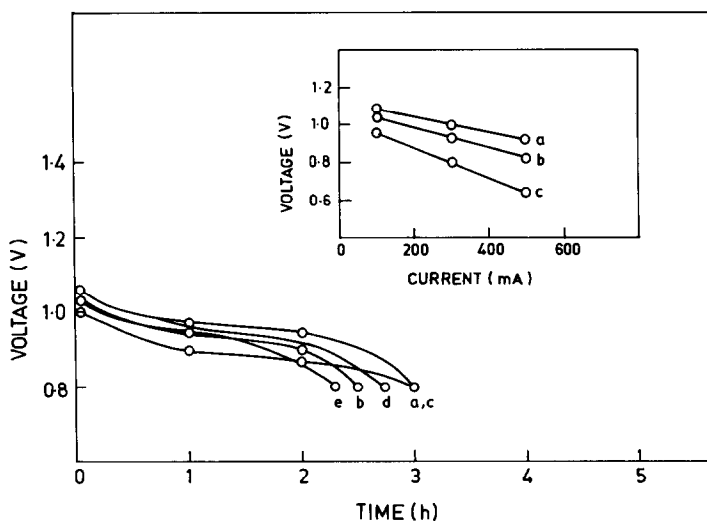


Fig. 8. Effect of temperature on  $\text{Mg}/\text{Mg}(\text{ClO}_4)_2/\text{CDNB}$  cells. Current drain: 200 mA, and temperature: +10 °C. (a) *m*-DNB alone; (b) CDBN alone; (c) CDBN + CuO; (d) CDBN +  $\text{Fe}_2\text{O}_3$ , and (e) CDBN +  $\text{V}_2\text{O}_5$ . Inset. Variation of voltage with current drain for  $\text{Mg}/\text{Mg}(\text{ClO}_4)_2/\text{CDNB}$  cells at different temperatures: (a) +10 °C; (b) 0 °C, and (c) -10 °C.

The peak current ( $i_p$ ) was found to increase with the sweep rate ( $\nu$ ), but the  $i_p/\sqrt{\nu}$  values were not constant. This indicates that the overall process is kinetically controlled. The possible chemical step that controls the overall process may be due to a slow protonation of CDBN before electron transfer in neutral magnesium perchlorate medium.

The peak potential was found to shift towards more negative potentials with increasing sweep rate and no anodic peak was observed on the reverse scan. This indicates that the reduction of CDBN is an irreversible process.

TABLE 4

Capacity, energy density and cathode efficiency of cells at different temperatures and  $4.2 \text{ mA cm}^{-2}$

Depolarizer	Temperature ( $^{\circ}\text{C}$ )								
	+10			0			-10		
	A	B	C	A	B	C	A	B	C
<i>m</i> -DNB	0.20	0.18	10	0.13	0.11	7	0.07	0.06	4
CDNB	0.17	0.16	10	0.11	0.10	7	0.05	0.04	3
CDNB + CuO	0.20	0.20	13	0.15	0.14	9	0.09	0.08	6
CDNB + $\text{Fe}_2\text{O}_3$	0.18	0.18	11	0.13	0.12	8	0.13	0.05	8
CDNB + $\text{V}_2\text{O}_5$	0.16	0.16	10	0.13	0.12	8	0.06	0.05	4

A = Capacity ( $\text{Ah g}^{-1}$ ).

B = Energy density ( $\text{Wh g}^{-1}$ ).

C = Cathodic efficiency (%).

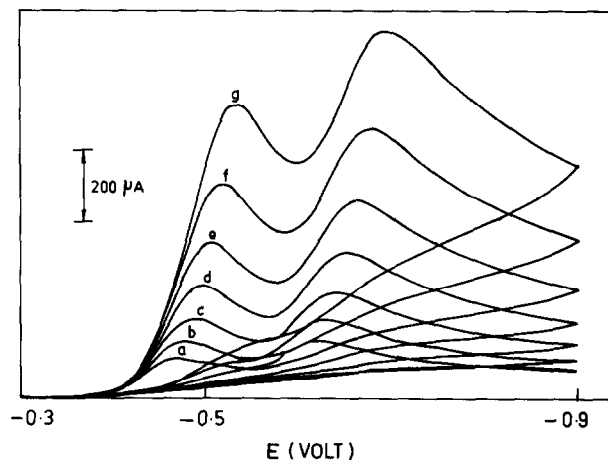


Fig. 9. Cyclic voltammograms for 10 mM CDNB at different sweep rates in 2 M  $\text{Mg}(\text{ClO}_4)_2$ . Scan rate ( $\text{mV s}^{-1}$ ): (a) 5; (b) 10; (c) 20; (d) 40; (e) 80; (f) 160, and (g) 320. Temperature:  $28 \pm 1$   $^{\circ}\text{C}$ .

Overall, cyclic voltammetric data suggest that the reduction process of CDNB in a neutral medium is a kinetically-controlled, irreversible process. Even though protonation is a slow reaction among the compounds compared here, protonation and reduction of CDNB is comparatively faster (see above). This is evident from the observed high capacity of the cells with CDNB as a depolarizer.

### Conclusions

1. The optimum percentage of conducting material (acetylene black) is 50 wt.%.
2. The operating voltage and reduction efficiency of the cell is higher for CDNB than for *m*-DNB. This is due to the substitution of a chloro group in the benzene nucleus.

TABLE 5

Cyclic voltammetric parameters obtained for the reduction of 10, 20 and 30 mM of CDNB at a glassy-carbon electrode in 2 M  $\text{Mg}(\text{ClO}_4)_2$  at  $28 \pm 1$  °C

Sweep rate ( $\nu$ ) ( $\text{mV s}^{-1}$ )	I peak			II peak		
	$i_p$ (mA)	$-E_p$ (V)	$i_p/\sqrt{\nu}$	$i_p$ (mA)	$-E_p$ (V)	$i_p/\sqrt{\nu}$
(a) 10 mM						
5	0.1160	0.471	0.0520	0.0740	0.620	0.0331
10	0.1610	0.478	0.0509	0.1030	0.632	0.0325
20	0.2180	0.488	0.0487	0.1350	0.639	0.0300
40	0.3050	0.498	0.0482	0.1490	0.652	0.0236
80	0.4320	0.505	0.0483	0.2100	0.664	0.0234
160	0.5960	0.518	0.0471	0.2950	0.678	0.0233
320	0.8200	0.532	0.0460	0.3700	0.694	0.0207
(b) 20 mM						
5	0.1910	0.476	0.0856	0.1200	0.637	0.0537
10	0.2600	0.485	0.0822	0.1600	0.643	0.0506
20	0.3250	0.497	0.0727	0.2100	0.656	0.0469
40	0.4880	0.506	0.0772	0.2900	0.669	0.0459
80	0.6510	0.518	0.0728	0.3700	0.682	0.0414
160	0.8900	0.532	0.0703	0.4980	0.700	0.0393
320	1.3280	0.550	0.0742	0.6140	0.721	0.0343
(c) 30 mM						
5	0.2590	0.481	0.1161	0.1500	0.644	0.0671
10	0.3560	0.490	0.1126	0.2120	0.654	0.0670
20	0.4840	0.502	0.1082	0.2740	0.666	0.0613
40	0.6660	0.516	0.1053	0.3640	0.682	0.0576
80	0.8840	0.529	0.0988	0.4750	0.699	0.0531
160	1.2080	0.546	0.0954	0.6120	0.721	0.0484
320	1.5230	0.564	0.0851	0.7360	0.745	0.0411

3. Addition of catalyst improves further the operating voltage and capacity of the cell. Among the catalysts added, CuO is found to yield the best performance: the Wh capacity is raised from 0.54 to 0.67 Wh  $\text{g}^{-1}$  at a current density of 4.2 mA  $\text{cm}^{-2}$  at  $28 \pm 1$  °C.

4. The cells exhibits very good low-temperature performance.

5. The reduction of CDNB is kinetically-controlled and irreversible.

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