

brass and coated by electrodeposition with the various metals.

The system was evacuated with a mercury diffusion pump in series with a rotary oil pump. A hydrogen pressure of approximately 0.5 mm. was maintained constant during each run as indicated by a McLeod gage.

In our measurements, the rise in temperature with time was recorded and the final steady-state temperature used in calculating results. Cobalt was chosen as the standard because of its high efficiency and resistance to poisoning. In each group of runs, the discharge was left on continuously and the cobalt cone and the others with which it was compared were introduced successively, each cone being introduced several times. The temperature rise relative to cobalt was thus obtained for each surface. Our results are summarized in Table I.

Freshly electroplated cones were used for each run. Although the absolute magnitude of  $\Delta V$  varied from run to run, the values relative to cobalt remained quite reproducible. On the basis of the preceding results, the surfaces investigated may be assigned an order of activity as follows

Pt	Cu	Zn	
Co	> brass	> paraffin	> Sn
Ni	Fe		
	Cr		
	Cd		

It was observed that the activity of a given cone tended to decline with time. This is probably attributable to progressive poisoning. The results were so reproducible that we believe the above relative efficiencies to be more accurate than those previously determined.<sup>1,2,3</sup>

(1) E. Boehm and K. F. Bonhoeffer, *Z. physik. Chem.*, **119**, 385 (1926).

(2) H. S. Taylor and G. I. Lavin, *THIS JOURNAL*, **52**, 1910 (1930).

(3) G. I. Lavin and W. F. Jackson, *ibid.*, **53**, 3189 (1931).

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### Dinitration of *p*-Bromotoluene

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The simple nitration of *p*-bromotoluene has been studied in detail<sup>1</sup> but more vigorous nitration leading to the introduction of two nitro groups has not been hitherto reported. In the present study, the only product successfully isolated was 2,3-dinitro-4-bromotoluene, which was synthesized independently from 2,3-dinitro-4-aminotoluene. The 2,5-dinitro isomer was also prepared, but from 2,5-dinitro-4-aminotoluene. Both isomers and also the 3,5-dinitro isomer were reduced to the amines and acetylated.<sup>2</sup>

#### Experimental

The melting points given are uncorrected.

(1) (a) Shaw and Turner, *J. Chem. Soc.*, 1884 (1932); (b) Holleman, *Rec. trav. chim.*, **34**, 283 (1915).

(2) Ipatieff and Schmerling, *THIS JOURNAL*, **59**, 1056 (1937).

**2,3-Dinitro-4-bromotoluene.**—One ml. of *p*-bromotoluene was added dropwise to a vigorously shaken mixture of 4 ml. of 98% sulfuric acid and 2 ml. of 70% nitric acid, the temperature rising to 90° and then subsiding. The semi-solid product was washed free of acid and crystallized from alcohol. About 200 mg. of colorless needles was obtained which melted at 129–130° after recrystallization from alcohol and which showed no depression in a mixed melting point determination with a sample prepared independently by the method described in the next paragraph.

One gram of 2,3-dinitro-4-aminotoluene<sup>3</sup> was slurried in 5 ml. of 98% sulfuric acid, cooled externally with ice, and 1 g. of sodium nitrite dissolved in 3 ml. of sulfuric acid was added slowly. The resulting slurry was stirred for one hour and a few crystals of sulfamic acid were then added. The slurry was added to a mixture of 25 ml. of 48% hydrobromic acid, 5 g. of cuprous bromide and 100 g. of ice. The mixture was allowed to stand in the cold for two days and was then poured into excess ice and filtered. The filter cake was leached with 50 ml. of hot alcohol, filtered and the filtrate evaporated to dryness. The residue was warmed on a steam-bath with 10 ml. of 70% nitric acid in which it dissolved. As soon as the evolution of nitric oxide ceased, the solution was diluted with a few ml. of water and cooled. The product formed colorless needles which when recrystallized from alcohol melted at 129°. The yield was about 200 mg.

*Anal.* Calcd. for C<sub>7</sub>H<sub>5</sub>O<sub>4</sub>N<sub>2</sub>Br: C, 32.2; H, 1.92; N, 10.72; Br, 30.6. Found: C, 32.0; H, 2.06; N, 11.0; Br, 30.6.

**2,5-Dinitro-4-bromotoluene** was prepared from 2,5-dinitro-4-aminotoluene<sup>3</sup> by the method described above. After two recrystallizations from alcohol it was obtained as straw-colored needles which melted at 111°.

*Anal.* Calcd. for C<sub>7</sub>H<sub>5</sub>O<sub>4</sub>N<sub>2</sub>Br: N, 10.72. Found: N, 10.9.

**2,5-Diacetamino-4-bromotoluene** was prepared by reducing the dinitro compound with tin and hydrochloric acid, followed by acetylation with acetic anhydride.<sup>2</sup> The product was twice recrystallized from alcohol and formed colorless needles which melted at 284°.

*Anal.* Calcd. for C<sub>11</sub>H<sub>13</sub>O<sub>2</sub>N<sub>2</sub>Br: C, 46.3; H, 4.56; N, 9.83; Br, 28.1. Found: C, 46.5; H, 4.70; N, 9.65; Br, 28.1.

2,3-Dinitro-4-bromotoluene failed to yield a crystalline derivative by this method.

**3,5-Diacetamino-4-bromotoluene** was prepared from 3,5-dinitro-4-bromotoluene<sup>4</sup> by the foregoing method. The product, recrystallized from alcohol, formed colorless needles which melted at 244.5–245°.

*Anal.* Calcd. for C<sub>11</sub>H<sub>13</sub>O<sub>2</sub>N<sub>2</sub>Br: C, 46.3; H, 4.56; N, 9.83; Br, 28.1. Found: C, 46.2; H, 4.64; N, 9.86; Br, 28.3.

(3) Page and Heasman, *J. Chem. Soc.*, **123**, 3235 (1923).

(4) Jackson and Ittner, *Am. Chem. J.*, **19**, 7 (1897).

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### *meso*-2,5-Dibromohexane

BY NATHAN KORNBLOM AND JOHN H. EICHER

The conversion of 2,5-hexanediol to 2,5-dibromohexane in 70–90% yields has previously been accomplished by treatment with fuming hydrobromic acid.<sup>1,2,3</sup> A distinctly more convenient procedure, which routinely gives better than 90% yields of 2,5-dibromohexane, involves treating the

(1) Duden and Lemme, *Ber.*, **35**, 1335 (1902).

(2) Pace, *Arch. farmacol. sper.*, **42**, 167 (1926); *C. A.*, **21**, 1964 (1927).

(3) W. H. Florsheim, Ph.D. Thesis, University of California at Los Angeles, May, 1948.