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N,N-DIMETHYLFORMAMIDE, A NEW DILUENT FOR  
METHYLENE IODIDE HEAVY LIQUID\*

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Dimethyl sulfoxide (DMSO),  $(\text{CH}_3)_2\text{SO}$ , has been recommended as a diluent for methylene iodide (diiodomethane) in heavy liquid separations (Cuttitta, Meyrowitz, and Levin, 1960). Dimethyl sulfoxide-methylene iodide liquids of low density (approximately 2.92) become blood red on long standing. Storing the liquids in contact with copper wire or shavings reduces markedly the rate at which the red color deepens.

N,N-Dimethylformamide (DMF),  $\text{HCON}(\text{CH}_3)_2$ , is recommended as a diluent for methylene iodide. Its physical properties are similar to dimethyl sulfoxide. Its vapor pressure is low; its boiling point is high and it is completely miscible with water and acetone. The advantage of the dimethylformamide liquids over the dimethyl sulfoxide liquids is that they remain transparent for a longer time. Mixtures of dimethylformamide and methylene iodide stored in contact with copper wire or shavings do not become red on long standing (seven months). They remain light yellow. They do become red when not stored in contact with copper wire or shavings. Table 1 compares the salient properties of dimethylformamide, dimethyl sulfoxide, acetone, ethyl alcohol, and methylene iodide.

In order to test the constancy of the dimethylformamide-methylene iodide solutions during use, a series of solutions, each having a 90 ml. volume, was prepared. The specific gravities of the liquids were 2.85,

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TABLE 1. SOME PHYSICAL PROPERTIES OF N,N-DIMETHYLFORMAMIDE, DIMETHYL SULFOXIDE, ACETONE, ETHYL ALCOHOL, AND METHYLENE IODIDE

	Melting point ° C.	Boiling point ° C.	Vapor pressure mm.Hg at 20° C.	Vapor pressure mm.Hg at 30° C.	Specific gravity	Flash point ° F.	Viscosity 25° C. cp
N,N-Dimethylformamide <sup>1</sup>	- 61	153	2.7	5.0	0.95	153	0.80
Dimethyl sulfoxide <sup>2</sup>	18.4	189	0.37	0.79	1.10	203	1.98
Acetone	- 95 <sup>3</sup>	56.5 <sup>3</sup> 185 <sup>4</sup>		283 <sup>4</sup>	0.79 <sup>3</sup>	15 <sup>3</sup>	0.32 <sup>3</sup>
Ethyl alcohol	-114 <sup>3</sup>	78.4 <sup>3</sup> 43.9 <sup>4</sup>		78.8 <sup>4</sup>	0.79 <sup>3</sup>	70 <sup>3</sup>	1.1 <sup>3</sup>
Methylene iodide	5-6 <sup>5</sup>	180 <sup>3</sup>	1.01 (20.6° C.) <sup>6</sup>	1.90 (29.5° C.) <sup>6</sup>	3.33 <sup>3</sup>	None	0.02 <sup>6</sup>

<sup>1</sup> Grasselli Chemicals Department, E. I. Dupont de Nemours and Co., Inc. DMF Product Information Bull., April 1, 1954.

<sup>2</sup> Stepan Chemical Co., Technical Bulletin, Dimethyl sulfoxide, Dec. 29, 1954.

<sup>3</sup> Hodgman, 1957.

<sup>4</sup> National Research Council, 1928.

<sup>5</sup> Gregory and Style, 1936.

<sup>6</sup> Timmermans and Hennaut-Roland, 1932.

2.91, 2.94, 3.06, 3.07, and 3.15. For each solution in the series, 12 to 16 heavy liquid separations, which included a filtration step, were made on approximately 10 gram portions of mineral mixtures over a period of 4 weeks. The remaining liquid (in contact with copper wire) was stored for an additional 6 months. The specific gravities of these liquids were determined again. The specific gravities of all of the liquids increased slightly.

Specific gravity before use	Specific gravity after use	$\Delta$ Specific gravity	Number of Separations
2.85	2.87	+0.02	16
2.91	2.94	+0.03	12
2.94	2.97	+0.03	14
3.06	3.09	+0.03	12
3.07	3.08	+0.01	15
3.15	3.17	+0.02	15

The combining volumes of dimethylformamide-methylene iodide solutions are additive and a straight-line mixing curve (volume+volume) can be used to prepare a liquid of desired specific gravity. Acetone, dimethylformamide, and water are miscible in all proportions. The separated minerals can be washed free of a dimethylformamide-methylene iodide liquid using acetone. The methylene iodide can be recovered from the washings by mixing the washings with large volumes of water in the manner conventionally used when alcohol or acetone is the diluent. When a dimethylformamide-methylene iodide solution, prepared for a specific job, is no longer needed, the methylene iodide can be recovered in the same way.

Information supplied by the manufacturers of dimethylformamide

states that toxicity does not seem to be a problem. Although the maximum allowable concentration in the atmosphere of dimethylformamide is relatively small, its "low volatility appears to make its use less hazardous from a vapor standpoint than that of many of the commonly-used organic solvents." It is suggested that contact of the dimethylformamide with the skin should be avoided and if it is spilled on the skin, it should be immediately flushed with a generous quantity of water. Breathing of the vapors should be avoided and adequate ventilation should be provided during use.

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PLANCHET PRESS AND ACCESSORIES FOR MOUNTING  
X-RAY POWDER DIFFRACTION SAMPLES

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Quantitative  $x$ -ray powder diffraction by the technique described by Klug and Alexander (1954) requires uniform packing of finely ground powder samples with minimal preferred orientation. Ideally the technique should be simple, fast, and reliable. Widely used mounts for powders are a smear on a glass slide and powder packed into depressions in glass slides (Klug and Alexander, 1954). Another approach to the problem of mounting samples is to sediment or press the powder into a mold, mount, or planchet (Adams and Rowe, 1954; Holland, *et al.*, 1955; and McAfee, 1956). All of the described devices had some shortcomings for our requirements. We therefore modified a pellet press\* to pack samples to meet our needs. A monel planchet is used to avoid iron fluorescence in Cu  $K\alpha$   $x$ -ray radiation.

\* Parr Instrument Co., Moline, Illinois.