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EFFECT OF MERCURY-ALLOY RATIO ON THE PHYSICAL PROPERTIES OF AMALGAM

by

W. T. Sweeney* Claire L. Burns**

- * Chief, Dental Research Section, National Bureau of Standards.
- ** Chemist, Dental Research Section, National Bureau of Standards.

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U. S. DEPARTMENT OF COMMERCE NATIONAL BUREAU OF STANDARDS

EFFECT OF MERCURY-ALLOY RATIO ON THE PHYSICAL PROPERTIES OF AMALGAM

Amalgam

Some physical properties of amalgams made from four brands of dental alloy were studied for mercury to alloy ratios from 1 to 1 to ratios of 10 to 1. The compressive strength, dimensional change on setting, flow and residual mercury content were determined by standards methods. For mercury-alloy ratios ranging from the manufacturers' recommended values to one part alloy per ten parts of mercury there was little observed effect on the compressive strength. Over this range of ratios the residual mercury content varied a maximum of 3% for any one alloy. An additional study was made of the effect of strain rate on crushing strength using 4 mm x 8 mm cylindrical specimens. Varying head speed from .003 to .050 inch per minute produced crushing strengths ranging from 30,000 to 50,000 psi. These data indicate that the physical properties of amalgam are not significantly affected by mercury-alloy ratio, provided an essential minimum of mercury is present.

I. INTRODUCTION

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Silver amalgam is the most important filling material as it is used in more tooth restorations than are all other materials. The dentist has a critical interest in how the mercury-alloy ratio effects the properties of the finished tooth restoration because he, in fact, fabricates the amalgam by the addition of the mercury to the silver-tin-copper-zinc powder he is furnished by the manufacturer. It has been reported by many workers that the properties of dental amalgam are dependent upon the amount of mercury in the condensed alloy, usually referred to as residual mercury. The unique method used in making dental amalgam is conducive to the production of a variable composition in the resulting filling. This is due to adding an excess of mercury, during mixing and subsequently removing part of it by expressing through a cloth and by rejecting part of the mercury-rich mass during packing.

Phillips and Swartz [1] reported values for the residual mercury content of 100 amalgam fillings removed from extracted teeth. The average mercury content was 45.4 percent, and the type of preparation had little effect on the final mercury content. Healey and Phillips [2] studied the cause of clinical failures and concluded that 40 percent of them are due to faulty manipulation and 26 percent to fractures. This points up the practical importance of knowing which factors effect the strength of the amalgam restoration. The present report is on a well controlled series of measurements on four widely used silver alloys considered representative of the type currently used in this country. They are on the American Dental Associations's List of Certified Materials. The compositions of the four alloys are similar, with the exception that one does

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not contain any zinc. However, the four are quite different in particle size. Alloys differing in particle size were selected so the information obtained would be applicable to amalgams in general use.

Eames [3] has reported very satisfactory clinical results on amalgam restorations that were mixed with a mercury to alloy ratio of about 1:1. He found high 1 hour compressive strength, approximately 40 percent of the one day strength. Taylor et al [4] reported about 15 percent of the one day strength at one hour. The rate of loading the specimens may be a factor in this variation as well as the mercury alloy ratio.

2. MATERIALS INVESTIGATED

The following materials were investigated:

Alloy	Manufacturer
Twentieth Centu r y Micro Non- zinc Pellets	The L. D. Caulk Co.
Silver Crown Medium	General Refineris, Inc.
Aristaloy	Baker and Co., Inc.
Twentieth Century Regular	The L. D. Caulk Co.

Batches of the above alloys made in 1957-59 had the analyses shown in Table I.

3. TEST PROCEDURE AND RESULTS

3.1 Compressive Strength

All the compressive strength specimens were triturated with a mechanical amalgamator (Wig-l-bug) for the time recommended by the manufacturer. Alloys 1 to 4 were mixed for

12, 8, 7 and 13 seconds, respectively. The mixes were made by placing 0.4 g of alloy with the predetermined amount of mercury in the capsule with the pellet. In most cases 2 or 3 mixes were necessary. Specimens were made using mercury to alloy ratios of 1 to 1, the manufacturer's recommended ratio, 4 to 1 and 10 to 1. The recommended ratios were 7 to 5, 9 to 5, 8 to 5 and 8 to 5, respectively, for the four alloys. All specimens were hand packed, following a uniform technic of removing the excess mercury from increments of the mixed amalgam by squeezing it through a cloth immediately before packing in a steel mold 4 mm in diameter by 8 mm long. The top of the specimen was cut off with a razor blade before removal from the mold. The packing was accomplished with a 1.5 mm plugger with approximately 4 to 6 pounds pressure. This corresponds to about 2000 psi. A larger plugger was used to remove excess mercury during packing. The rate of loading was studied for head speeds of 0.003, 0.010, 0.025, and 0.050 in./min using the Instron Testing Machine which applies a strain at a uniform rate. Also, for comparison, data were obtained using the Tinius Olson pendulum type machine at a loading rate of 100 pounds per minute. The results are shown in Table II and Figures 1, 2 and 3. The data reported are the average of six specimens in all cases and the standard deviation is indicated to show the spread of the results. The results reported are for specimens 24 hours old.

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3.2 Flow

The flow specimens were prepared by the same technic as the compressive strength specimens described, and the tests for the measurement of flow were conducted as described in the ADA Specification No. 1 for Dental Amalgam Alloy. The values reported in Table III are for duplicate specimens for the same mercury to alloy ratios, as for compressive strength.

The data indicate that the flow was slightly higher for the 1 to 1 ratio, but on the average the flow is independent of the mercury-alloy ratio.

3.3 Setting Change

The setting change was measured on the four mercury to alloy ratios for both mechanically and hand triturated specimens. The results are shown in Table IV and Figures 4 to 9. The tests were made according to ADA Specification No. 1 for Dental Amalgam Alloy. All specimens were packed by hand using the same technic as outlined for making the compressive strength specimens.

The mechanically mixed specimens expanded less in most cases than the hand mixed specimens. All results on hand triturated specimens were within the Specification limits of 3 to 13 microns per centimeter, while the values for the mechanically mixed ratios were in most cases below the limit except for the highest mercury to alloy ratio. Alloy 1 did not seem to follow this trend for

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the 10 to 1 ratio. It is considered that these rather small variations are of little clinical significance.

3.4 Residual Mercury

The method for analyzing the mercury content of the amalgam was that of Crawford and Lawson [5]. The values reported are for the mechanically mixed, compressive strength specimens after crushing. Duplicate tests were made on each specimen. The recorded values for each ratio being the average of twelve separate determinations of the mercury content. (Table V and Figure 10). The average variation as measured by the standard deviation is about 0.7 percent. The maximum difference in mercury is 3.2 percent for any individual alloy, and in almost all cases the mercury was higher in the amalgam with the greater additions of mercury to the mix. The average mercury content of the four amalgamswas 48.5 percent.

4. DISCUSSION

The flow results on the four alloys indicate that the mercury-alloy ratio has little effect on this property. The flow of specimens with the 1 to 1 ratio is slightly higher for most of the alloys. The flow can be interpreted as an index to the early strength for the various mercury-alloy ratios. The major factor in this type of flow test is resistance to dimensional change under load soon after the specimens are placed in the equipment. Inasmuch as the test load is applied at 3 hours after mixing all the specimens must by necessity

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have hardened, or developed considerable strength by that time.

The four alloys when triturated by hand at mercury-alloy ratios between 1 and 10 meet the setting changes requirement of ADA Specification No. 1 for Dental Amalgam Alloy. The mechanically triturated specimens, in general, gave lower results, being below the specification requirements for the 1 to 1 and the manufacturers' recommended ratios. Conversely, mechanically triturated specimens with the higher mercury-alloy ratios are generally within the specification limits. However, variations of a few microns per centimeter are considered by the authors to be of little clinical significance.

The amount of residual mercury was larger in the higher mercury-alloy ratios. However, the average deviation for any of the individual alloys is within plus or minus 1 percent. This small difference in mercury content cannot be correlated with the physical properties measured. The data show that a good technic for packing these alloys resulted in consistently uniform amalgam for mercury-alloy ratios of from 1 to 1 to ratios of 10 to 1. The results indicate that the method of packing and mixing technic may be more important than the mercuryalloy ratio used.

The values for compressive strength are dependent on the rate of strain of the specimens. A change in head speeds from 0.003 to 0.050 inch per minute gives an increase in compressive strength in the order of 50 percent. This means that

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it is essential to know the rate of strain when reporting the strength of amalgam alloys. The values for the strength of the materials tested vary with the log of the strain rate as shown in Figure 2.

A resume of the effect of mercury-alloy ratio is shown in Table VI which gives the relative strength for a typical set of data, 0.003 inch per minute, using the recommended ratio as the basis for comparison. The percentage change is not more than 7 percent for the mercury-alloy ratios greater than those recommended by the manufacturer. The variations in strength for all ratios are in general, within the experimental error. This indicates that the strength is independent of the mercury-alloy ratios tested.

5. SUMMARY

Data on flow, setting change, strength, rate of loading, and mercury content for four typical amalgam alloys are reported. The mercury-alloy ratio is shown to have very little effect on the physical properties investigated for ratios equal to or greater than the manufacturer's recommended ratio. The compressive strength at 24 hours was found to be only a few percent different from the manufacturer's recommended ratio to a 10 to 1 ratio for the technic employed. The flow results all fall within the limits of ADA Specification No. 1 with the exception of those for alloy 1 at the 1 to 1 ratio. All hand triturated setting change results meet the requirements of the same specification. Mechanically triturated setting

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change specimens are somewhat below the limits for the lower ratios. The clinical significance of the results is of considerable importance as they would indicate that it is not necessary to determine precisely the amount of mercury to be mixed with the alloy provided there is an amount equal to or in excess of the manufacturer's recommended ratio. Care must be taken to adequately express the excess mercury and to firmly condense the amalgam.

REFERENCES

- Phillips, R. W. and Swartz, Marjorie L. Mercury analysis of one hundred amalgam restorations.
 J. D. Res. 28:569 Dec. 1949.
- Healey, H. J. and Phillips, R. W. A clinical study of amalgam failures. J. D. Res. 28:439 Oct. 1949.
- 3. Eames, Wilmer B. Preparation and condensation of amalgam with low mercury-alloy ratio. JADA 58:78 April 1959.
- 4. Taylor, N. O., Sweeney, W. T., Mahler, D. B. and Dinger, E. J. The effect of variable factors on the strength of dental amalgam. J. D. Res. 28:228 June 1949.
- Crawford, W. H. and Larson, J. H. Residual mercury determination process. J. D. Res. 34:303 June 1955.

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Table I

Analysis of Silver Alloys for Dental Amalgam

Alloy*	Silver	<u>Tin</u>	Copper	Zinc	Remarks
1	70.0%	26.3%	3.8	%	Pellets
2	70.0	26.6	2.6	0.8	Medium
3	69.0	26.7	3.8	0.5	Fine
4	70.0	26.0	3.5	0.5	Regular

* Analysis on batches obtained 1957-59.

Table II

Compressive Strength of Amalgams

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	4	N N • 8 1 • 1	ноног		N N N F	a wh	4021 115
		C.S. psi*	10000 000000 000000	10.2	44 44 44 44	04-1-0 04-1-0	500 11300 11300
	~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	S.D. psi*	4000H		5000 1000	~~~~ ~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	1.1 0.6 0.6
	(*)	d.S. Ssit.∗	000000 0000000000000000000000000000000	0.4 V	0 m 0 0 m 0	51 44 50 6	38.5 40.1
loy		U M				-1-0101	(1)
ΑJ		S.D. psi*	10000 70000	110 100 100	<b>00</b> 20	0 H M M M H	1.4 0.6 0.7
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loy	C H		U	U	U	U	U
Ig/Al	ה שנו		H B H	Re L Re	Re L Re L	Re 1 te	Re Re
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psi in 1000. Tinius Olson testing machine, 100 lbs/min.

Specimen age: 24 hours.

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Effect of Mercury-Alloy Ratio on Flow of Amalgam

	Alloy						
Hg/Alloy	1	2	3	4	Average		
l	5.0%	2.2%	3.6%	2.5%	3.3%		
Rec	3.2	1.3	2.4	2.8	2.4		
4	3.8	1.8	1.8	2.0	2.4		
10	2.8	1.5	2.2	1.5	2.0		
Avg.	3.7	1 <b>.</b> 7	2.5	2.2	2.5		

Table IV

Effect of Mercury-Alloy Ratio on Setting Change

Havallow	Alloy								
IIG/AIIOy		1		2		3			4
	Mech.	Hand	Mech.	Hand	Mech.	Hand	Ме	ch.	Hand
	u/cm	u/cm	u/cm	u/cm	u/cm	u/cm	u/	′cm	u/cm
1	- 8	10	2	5	2	12	and a subscript of the subscript of	6	7
Rec	-12	6	-2	4	0	6		-7	4
4	3	10	0	5	4	8		2	6
10	14	13	4	4	8	9		4	8

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Effect of	Mercury-Allo	y Ratio on	Residual	Mercury
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	Alloy								
Hg/Alloy		1		2		3	2	Ì	
	%	S.D.	%	S.D.	%	S.D.	%	S.D.	
1	48.4	1.3	46.4	0.5	45.8	0.8	48.4	0.4	
Rec	48.8	0.2	47.7	0.6	45.5	0.4	49.4	0.4	
2	49.8	0.7	47.2	0.6	46.3	0.9	49.2	0.5	
4	50.4	0.9	48.6	0.8	47.5	0.8	50.1	0.4	
10	51.6	0.3	48.6	1.4	48.5	0.7	51.6	1.4	

## Table VI

	Alloy							
Hg/Alloy	1	2	3	4				
	%	%	%	%				
1	92	97	88	86				
Rec	100	100	100	100				
2	101	99	101	96				
4	104	101	100	97				
10	107	99	99	100				

Strength of Amalgam

Head speed: 0.003 in./min.

Specimen age: 24 hours.







Figure 2































