LA-UR -81-917

CONF-810602--11

TITLE: EUTECTIC COMPOSITE EXPLOSIVES CONTAINING AMMONIUM NITRATE

AUTHOR(S): Mary M. Stinecipher

MASTER

SUBMITTED TO: 7th Symposium (Intl) on Detonation
Annapolis, MD

University of California

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EUTECTIC COMPOSITE EXPLOSIVES CONTAINING AMMONIUM NITRATE

M. M. Stinecipher
Los Alamos National Laboratory
University of California
Los Alamos, New Mexico

We have prepared, and studied the sensitivity and performance of, the eutectic of ammonium nitrate (AN) and the ammonium salt of 3,5-dinitro-1,2,4-triazole. We found that this AN formulation was unusual in that it performed ideally at small diameter, which indicated that it was a monomolecular explosive. Sensitivity tests included type 12 impact, Henkin thermal and wedge tests, and performance tests included rate stick/plate dent, cylinder, and aquarium tests. Results were compared with calculations, standard explosives, and another eutectic, ethylenediamine dinitrate (EDD)/AN.

I. INTRODUCTION

Monomolecular explosives are limited in amount of energy that can be built into the molecule without it becoming too unstable to use. However, if all the oxygen does not have to be in the same molecule, a more stable combination of oxidizer and fuel-rich explosive can achieve the energy of a CO_2-balanced explosive. Until recently, it had been thought (1) that a solid solution would be necessary for an oxidizer and fuel to behave as one compound in the detonation zone. However, experiments by Finger (2) and co-workers at Lawrence Livermore National Laboratory (LLNL) showed that when the particle size of the oxidizer was less than 5 μm (for example, a composite of ground particles of ammonium perchlorate suspended in gelled nitromethane) then the oxidizer reacts completely in the detonation zone, as shown by the scaling between 2.54- and 5.08-cm-diam cylinder tests. McGuire (3) found that isotopic mixing during detonation in a monomolecular explosive was random. But in the case of a composite, Amatol (N^{15}-labeled ammonium nitrate (AN)/TNT) the isotope ratio in the products indicated that only a 0.6 μm layer of the AN particles combined with the atoms of TNT in the detonation zone before the products were frozen out (3). These two experiments show that if the particle size of the oxidizer is less than about 1 μm, a solid solution will not be necessary to get complete reaction in the detonation zone.

Akst and Hershkowitz found that cosolidification of AN with other amine nitrates increased the participation of AN in the detonation. They found the eutectic of AN with ethylenediamine dinitrate (EDD) especially interesting because its melting point at 103°C would make it a useful casting matrix (4,5,6). They found some improvement in the amount of AN reacting in the detonation zone, but their experiments with EDD/AN at Picatinny Arsenal (now U.S. Army Armament Research and Development Command, ARRADCOM) were limited to small diameter detonation velocities (D). They did not come close to calculated values of D at these diameters.

At the same time at Los Alamos National Laboratory, we began to study the eutectics of AN with the ammonium salts of nitroheterocycles. One of these, the eutectic of AN with the ammonium salt of 3,5-dinitro-1,2,4-triazole (ADNT) was stable as a melt from 112 to 130°C, and needed only two moles of AN per mole of ADNT to be CO_2-balanced. Because the CO_2-formulation was not far from eutectic formulation, 1.38/1 molar ratio AN/ADNT, all of the AN could be dissolved in the eutectic melt by heating to 123°C (7). We have studied the performance of the eutectic and 2/1 molar ratio AN/ADNT, compared these results with calculated results and compared our results with those Akst has obtained for eutectic EDD/AN (8).

II. MATERIAL PREPARATION

Preparation of ADNT

We have modified the method of preparation of ADNT reported by the Soviets (9), especially the method of isolation. The following is a sample synthesis and isolation.

A solution of 3,5-diamino-1,2,4,- triazole (Aldrich) (30.0 g, 0.3 mol) in 0.68 M sulfuric acid (1.1 ℓ, 1.5 mol H^+) was added dropwise over 3 hours to a solution of sodium nitrite (200 g, 2.9 mol) in water (200 ml) while keeping the reaction mixture at 0°C with an alcohol-ice bath. After addition was complete, the reaction was heated to 60°C and was held there for one hour until all precipitate had dissolved. Then the reaction mixture was cooled again to 0°C, and acidified with 6 M sulfuric acid (100 ml, 1.2 mol H^+). This step converted the excess sodium nitrite to nitrous acid. Urea (15 g, 0.25 mol) was added slowly (foaming) to remove any remaining oxides of nitrogen from solution. The completed reaction mixture had a small amount of orange precipitate that was removed by addition of decolorizing carbon and filtration. Analysis of the filtrate by ultraviolet spectroscopy at 285 nm showed a 90% yield of 3,5-dinitro-1,2,4-triazole (DNT).

The strongly acidic character of DNT (-0.66 pKa) (10) made extraction with the usual organic solvents impossible. It can be extracted by the continuous method with ethyl ether, but the flammability of ether made another method necessary for large-scale reactions. We found that a toluene solution (20%) of water-insoluble secondary or tertiary amines extracted the DNT from the aqueous reaction mixture (11). Ammonia gas bubbled into the dried organic phase regenerated the amine and precipitated the desired ammonium salt, ADNT. Recrystallization in 90/10 volume ratio ethyl acetate/acetone and drying over Drierite in vacuum gave pure ADNT (35 g, 0.2 mol, 67% yield) with a melting point of 168-170°C. The yield was improved by a second extraction of the aqueous reaction mixture. ADNT forms a dihydrate at relative humidities over 35%. The amines used were tertiary amines: Alamines 304 and 336 (General Mill Chemicals, Inc.) and a secondary amine: Amberlite (Rohm and Haas Co.). The molar amount of amine used was equal to the amount of the DNT in the solution as measured by uv spectroscopy. The regenerated amine solution was reused after washing with water.

Preparation of Amine Nitrates

We prepared EDD and other aliphatic amine nitrates by dissolving the amine in 10% aqueous methanol and adding 50/50 by volume concentrated nitric acid/water dropwise while cooling with an ice bath to keep the temperature at ambient or below. After neutralization was complete, the precipitated nitrate salt was filtered, washed well with methanol, and recrystallized from water/methanol.

Preparation of AN/ADNT Mixes

Dry AN and ADNT (overnight in a vacuum desiccator over Drierite) were mixed and melted in a beaker heated by a heating bath or a heating block with a temperature between 120 and 135°C until a clear, molten solution was obtained. If a slurry of another explosive or additive was desired, it was added and mixed until uniform. The mixture was poured and spread onto a thin sheet on Teflon. While cooling, the sheet went through a pliable stage to a brittle stage. The cooled sheet was ground and pressed at 60 to 100°C under vacuum into cylindrical charges.

III. TESTING METHODS

Sensitivity Tests

Mixtures were tested for safety by finding impact and thermal sensitivities. The wedge test gave additional information on initiation.

Impact sensitivity was found on an ERL-type machine equipped with type 12 tools and a microphone for sound analysis. A 40 mg sample heaped on a square of sandpaper was placed under an anvil and a 2.5 kg weight was dropped onto the anvil. No sandpaper is used for type 12B. An arbitrary level of sound indicated a "go." The 50% "go" height was determined by the Bruceton Up and Down method (12).

The thermal sensitivity was measured by the modified Henkin method as developed by Rogers (13) and Janney. A thin slab of explosive was confined in a cartridge, placed in a temperature controlled bath of Wood's metal, and timed until explosion. The temperature below which it will not explode is the critical temperature.

The wedge test, a measure of distance of run of a shock before detonation starts in an explosive, was done at Los Alamos by the Detonation Physics group (14) on 30°-angle mini-wedges machined from 2.54-cm-diam pressed cylinders. Three pressures were used to find the relationship of the run to detonation, X*, to the pressure input.

Performance Tests

The detonation velocity was measured by two groups at Los Alamos. The method used by the Detonation Physics group has been described by Engelke (15); it is very accurate. The Los Alamos Explosives Technology group's method follows.

Ionization switches made of polyamide-insulated, four-mil copper wire were placed about 2.54 mm apart between the charges. The passage of the ionized detonation wave triggered a timer, accurate to ± 10 nanoseconds. We used a stack of six 2.54-cm-diam charges held tightly in place on a 5.08-cm-thick steel plate with tape. The dent was measured by a ball and ring method. Plate dents of the standard explosives have been correlated by Smith (16) and Urizar. For this 2.54-cm-diam unconfined plate dent test, a factor of 5.68 multiplied by the dent (mm) gave the detonation pressure (GPa).

Cylinder tests were done by the Los Alamos Detonation Physics group. The samples were fired in a 2.54-cm-inner-diam by 30.48-cm-long copper tube or a 5.08-cm-inner diam by 60.96-cm-long copper tube. A streak camera recorded the expansion of the copper at a slit 20.32 cm down the tube and appropriately scaled in the 5.08-cm tube. The time versus expansion data were analyzed by fitting a power curve through 30 points surrounding the 6-, 19-, and 30-mm points of expansion and by finding the first differential for velocity and the second differential for acceleration at each point (8). Energy of the expansion was compared to TNT by dividing the square of the velocity by the square of the velocity of TNT at the same expansion.

The aquarium test to compare the shock wave in water to what would be calculated was fired at Los Alamos by the Detonation Physics group with calculations done by the Detonation Theory and Application group (17).

The heat of detonation and products were determined in the calorimeter at LLNL as described by Ornellas (18).

IV. RESULTS

Sensitivity

We found that the impact and thermal sensitivities showed AN/ADNT and EDD/AN eutectics to be moderately insensitive and safe for most processes. See Table 1 for a comparison of the formulations tested with standard explosives. Note the critical temperature of AN/ADNT with nitroguanidine (NQ), which is higher than either AN/ADNT or NQ.

The wedge test showed that the shock sensitivity crosses the value of Composition B at a pressure of 6.8 GPa, but because the slope is different, it will have a longer run at lower shocks and a shorter run at higher shocks. It extrapolates to very small runs to detonation at 15 GPa. (Table 2 and Figure 1).

Performance

Initial plate dent tests on AN/ADNT mixtures were done in 1.27-cm-diam because of the limited supply of ADNT (Table 1). We were surprised that formulation containing almost 50% AN sustained detonation in a 1.27-cm-diam charge that was unconfined and gave a good dent. The pressure derived from dents was compared to that calculated by C. L. Mader of Los Alamos with the BKW hydrodynamics computer code (19). The 1.27-cm size was close to the failure diameter (one failed at high density) so subsequent tests were performed on 2.54-cm-diam charges.

TABLE 1

Sensitivity Tests

Material (molar ratios)	Impact Sensitivity Type 12 (cm)	12B (cm)	Critical Temperature Henkin (°C)
ADNT	58	96	225
ADNT $2H_2O$	224	>320	---
AN	>320	>320	361
1.38/1-AN/ADNT	66	71	236
2/1-AN/ADNT	65	65	241
2/1/1.3-AN/ADNT/TATB	250	270	236
1.38/1/1.54-AN/ADNT/RDX	37	70	215
5/1/1-AN/ADNT/RDX	44	74	219
5/1/1/3.3-AN/ADNT/RDX/Al	38	55	221
EDD	88	115	245
2.33/1-AN/EDD	109	153	244
3.7/1/1-AN/ADNT/EDD	93	99	212
3/0.3/1-AN/ADNT/EDD	58	96	227
FDEK	74	76	226
1.38/1/1.38-AN/ADNT/NQ	108	210	256
1.38/1/1.83-AN/ADNT/NQ	312	>320	261
NQ	>320	>320	198
RDX	22	30	214
TNT	150	>320	283

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where:

AN = ammonium nitrate
ADNT = ammonium dinitrotriazolate
EDD = ethylenediamine dinitrate
NQ = nitroguanidine
RDX = cyclotrimethylene trinitramine
TATB = 1,3,5-triamino-2,4,6-trinitrobenzene
TNT = 1,3,5-trinitrotoluene
Al - aluminum
FDEK = 2.55/0.3/1/0.36 - AN/ADNT/EDD/Potassium Nitrate (KN)

TABLE 2

Wedge Test Data on 2/1 - AN/ADNT

	First Shot	Second Shot	Third Shot
Density (g/cm^3)	1.640	1.640	1.640
Input Pressure (GPa)	6.9	8.9	7.8
Run to Detonation (mm)	6.1	2.7	4.8
Time (μs)	1.28	0.55	0.90

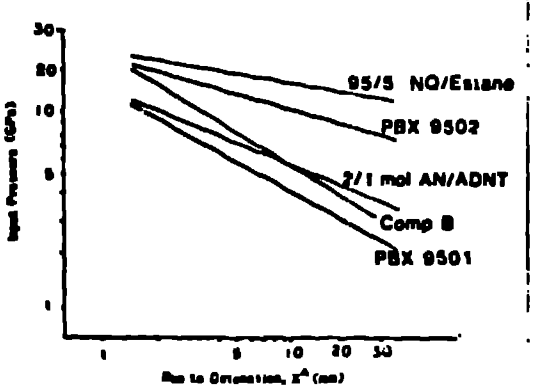

Fig. 1

Relative Shock Sensitivities of 2/1 AN/ADNT and Other Explosives in the Wedge Test.

[A] PBX 9501 - 95/2.5/2.5 wt% HMX/Estane/-NP
PBX 9502 - 95/5 wt% TATB/Kel-F 800

Later, plate dent tests were done in conjunction with detonation velocity tests on 2.54-cm-diam charges (Table 4). We found that the addition of extra AN to the eutectic formulation reduced the agreement with the calculated values with the exception of the aluminized 2/1 molar ratio AN/ADNT, which performed better than calculated.

An aquarium test on the 1.38/1/1.5 molar ratio AN/ADNT/RDX showed that the experimental shock in water was the same as the calculated pressure profile (Figure 2).

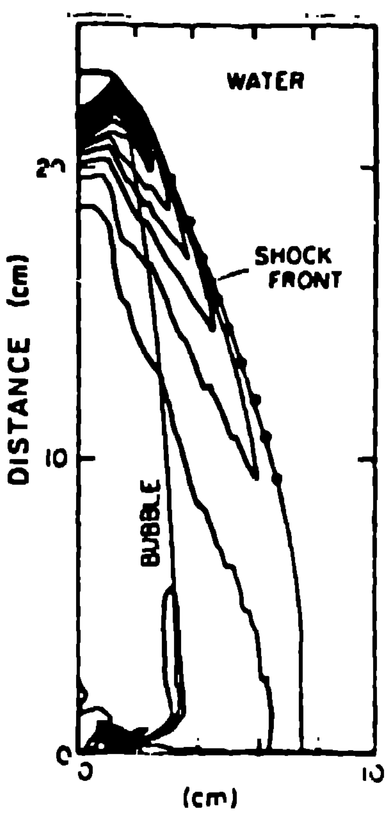

Fig. 2

Aquarium test of 1.38/1/1.5 AN/ADNT/RDX. The lines are the computer-generated shock-wave curves and the points are the experimental data.

TABLE 3

Unconfined 1.27 cm Plate Dents

Material (molar ratio)	Density (g/cm^3)	Extrapolated P_{CJ} (GPa)	Calculated P_{CJ} (GPa)
2/1-AN/ADNT	1.590	25.2	25.4
	1.645	23.0	27.6
1.38/1-AN/ADNT	1.630	27.3	26.7
	1.610	Failed	26.7
1.38/1/1.38-AN/ADNT/NQ	1.654	26.1	28.0
1.38/1/1.5-AN/ADNT/RDX	1.708	33.6	30.4
1.38/1/1-AN/ADNT/HMX	1.756	34.2	32.4

TABLE 4

Unconfined 2.54-cm Rate Stick/Plate Dent Tests

Material (molar ratio)	Density (g/cm^3)	Experimental P_{CJ}(dent) (GPa)	Experimental D(m/s)	Calculated P_{CJ}(GPa)	Calculated D(m/s)
2/1-AN/ADNT	1.640	26.1	7892±1	27.3	8268
1.38/1/1.5-AN/ADNT/RDX	1.717	31.7	8455±2	30.6	8475
5/1/1-AN/ADNT/RDX	1.699	24.0	7712±1	30.4	8598
5/1/1/3.3-AN/ADNT/RDX/Al	1.752	25.0	7739±10	29.1	8223
2/1/2.66-AN/ADNT/Al	1.734	26.3	7844	25.1	7680
2/1/1.3-AN/ADNT/TATB	1.765	28.3	7845±1	28.0	8013
1.38/1/1.38-AN/ADNT/NQ	1.655	26.4	------	28.0	8305
1.38/1/1.83-AN/ADNT/NQ	1.654	25.5	8160	26.8	8197
3/1/1-AN/ADNT/EDD	1.607	24.2	7664±1	27.4	8287
3/0.3/1-AN/ADNT/EDD	1.535	20.8	------	23.0	7870

The cylinder tests showed that the ability to push metal of the eutectic explosives was better than TNT (Table 5). These tests showed the effect of diameter on the behavior of intermolecular explosives. Comparison with the EDD/AN 5.08-cm and 10.16-cm cylinder tests showed their similarity in performance.

With detonation calorimetry we found that the heat of detonation and the products of 2/1 molar ratio AN/ADNT were what would be expected from a monomolecular compound (Table 6) from a BKWR Tiger calculation at freeze-out temperatures of 1500°K and 1800°K.

TABLE 5

Cylinder Tests

	TNT	ADNT	AN/ADNT	AN/ADNT/Al	AN/ADNT/Al	AN/ADNT/Al	EDD[a]	EDD/AN[a]	EDD/AN[a]
	2.54 cm	2.54 cm	2.54 cm	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
Density (g/cm^3)	1.61	1.58	1.610	[illegible]	1.613	[illegible]	1.551	1.576	[illegible]
Det. vel. (m/s)	6,950	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
Cylinder Wall Velocity (mm/μs)									
at 6 mm	1.23	1.10	[illegible]	[illegible]	1.27	[illegible]	1.10	[illegible]	[illegible]
19 mm	[illegible]	1.19	[illegible]	1.51	[illegible]	[illegible]	[illegible]	1.45	[illegible]
10 mm	1.44	1.53	(1.57)[b]	1.61	1.57	[illegible]	[illegible]	1.50	1.51
Cylinder Energy Relative to TNT v^2/v^2 TNT									
at 6 mm	1.00	1.12	1.25	[illegible]	[illegible]	[illegible]	1.12	[illegible]	1.19
19 mm	1.00	1.11	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	1.11
10 mm	1.00	1.10	(1.16)[b]	[illegible]	1.08	1.11	[illegible]	[illegible]	1.10

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[a] I. B. Akst [8]

[b] Expansion to this point was not filmed. This number was projected by averaging the expansion increment of ADNT, EDD, and the 10 cm EDD/AN cylinders.

AN/ADNT is the 2/1 molar ratio of [illegible]/52.[illegible] weight ratio mixture

AN/ADNT/Al is the 2/1/2.66 molar ratio of 39.2/43.2/17.6 weight ratio mixture

EDD/AN is 1/2.13 molar ratio or 50/50 weight ratio mixture

TABLE 6

The Detonation Calorimetry Test of 2/1-AN/ADNT

Density was 1.639 g/cm^3		Experimental	Calculations (LLNL)	
$-\Delta H$ detonation (cal/g)		1213±12	1263±40 (from products)	
Products (mol/mol HE)			BKWR CJ Isentrope	
			1520K	1825K
H_2O	1.78		1.77	1.78
N_2	1.47		1.49	1.49
CO_2	0.59		0.59	0.59
H_2	0.0096		0.0032	0.0025
CO	0.0092		0.0061	0.0065
NO	0.0013		0	0
NH_3	0.0006		0.0008	0.010
HCN	0.0005		0	0

V. DISCUSSION

Most explosives containing AN have large critical diameters and even larger ideal diameters. For example, 50/50 AN/TNT (density of 1.53 g/cm^3) has a critical diameter of 3.5 cm where D is 4500 m/s and an ideal diameter of 13 cm where D is 6300 m/s. The particle size, the density, and the degree of confinement affect the two diameters (18). Campbell and Engelke (15) reported the slowing of D as the diameter of the explosive approached the critical diameter. The amount of curvature is characteristic of the explosive, influenced by the reaction zone length.

The behavior of eutectic AN/ADNT is different because the critical diameter is about 1 cm and the ideal diameter about 2.54 cm. (D of unconfined rate stick and confined cylinder test of 2/1-AN/ADNT are the same; see Table 3 and 4.) D is about what would be expected from calculation (19) (Table 3).

This small ideal diameter is not found in every eutectic. Akst (3) has found that there was considerable difference in D of EDD/AN confined at 2.54-cm-diam, 6500 m/s, and at 10.16-cm-diam, 7410 m/s. We have compared the particle size of each eutectic by scanning electron microscopy and find that the AN particles in EDD/AN have a width of about [illegible] μm whereas the AN particles in AN/ADNT eutectic average 1-3 μm. As the ratio of AN/ADNT gets further from eutectic formulation, the particle size of AN increases to the same size as the AN in EDD/AN.

Another difference in the properties of the two eutectics occurs in the heat of formation. ADNT has a more positive heat of formation, 0.6 ± 2 kcal/mol, than EDD, -156.1 kcal/mol. That gives AN/ADNT more chemical energy to release during detonation, 1.10 kcal/g versus 0.95 kcal/g for eutectic EDD/AN (water as gas).

We have not determined which factor is more important or whether there is some other factor, which is not obvious now, that makes the difference. There are projects in progress to find ways to decrease the particle size of the AN in EDD/AN mixtures and search for other explosives that form eutectics with AN that would have different heats of formation.

Another way to increase the temperature in the detonation zone is to add aluminum metal powder. This should help increase the ideality of the performance because the temperature of the detonation affects the layer thickness of a particle of AN that will react in the detonation zone (19). We see that this is the case when going from the mixture 5/1/1 molar ratio AN/ADNT/RDX to one containing aluminum, 5/1/1/3.3 molar ratio AN/ADNT/RDX/Al. The detonation velocity increased from 90% to 94% of the calculated value. Addition of aluminum to 2/1 molar ratio AN/ADNT increased D from 95% to 102% of the calculated value. We tested the latter composition in the 2.54-mm-diam cylinder test. We found, as did Finger (20) and Bjarnholt (21), that aluminum behaves as an inert in the early part of the expansion (6 mm) but keeps the wall accelerating even at 30 mm. The wall velocity is almost as fast at 19 mm and is faster than the projected value of the 2/1 molar ratio AN/ADNT at 30 mm (Table 4). The projected value was determined by averaging the increments of the other cylinder tests that have D almost as calculated. The tests with D considerably less than calculated showed a higher increase in wall velocity from 19 to 30 mm than those with D close to calculated.

We have discussed ideality as the ability of an intermolecular explosive to follow calculated performance as do the monomolecular explosives that were used to calibrate the calculations. In the BKW calculations the agreement is not too good unless the ratios of the elements making up the new explosive are almost the same as in the model compound, RDX. Many of the intermolecular explosives have a much higher ratio of hydrogen to carbon than does RDX so they probably would not fit the calculation as well. For example, the RDX has C/H/O atom ratio 1/2/2 and 1.38/1 AN/ADNT has C/H/O atom ratio 1/4.76/4.07 but 2.33/1 molar ratio AN/EDD has C/H/O atom ratio 1/9.64/6.48.

Another way to look at ideality is by measuring the detonation products and comparing the result with what would be expected for complete reaction before freeze-out. The detonation calorimetry experiment (Table 6) showed that the products from the detonation of 2/1 molar ratio AN/ADNT, the CO_2-balanced intermolecular formulation, were what was expected. The oxygen from the AN has been used by the C or CO from the ADNT to form CO_2 almost exclusively. The recovery of the products was excellent because there were no solid products. There is definitely complete reaction in this case before freeze-out of the products.

As we study these eutectic systems and find others, we will better understand the parameters that affect the reactions between molecules in the detonation zone.

ACKNOWLEDGEMENTS

The author greatly appreciates the contributions in technical help and understanding given by Marion L. Clancy, M. L. Clancy, C. E. Hannaford, W. P. Fox, Joan L. Janney, M. J. Urizar, H. H. Cady, M. D. Coburn, Kien Y. Lee, R. P. Engelke, A. W. Campbell, C. W. Mautz, J. B. Ramsey, C. L. Mader, B. G. Craig, and especially the support by R. N. Rogers and conversations with I. H. Akst.

The work was supported in part by the Naval Sea Systems Command and Naval Surface Weapons Center block program under Department of Energy contract W-7405-ENG-36.

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