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AEC RESEARCH AND DEVELOPMENT REPORT

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From: R. E. Tomlinson

UNUSUAL INCIDENT AT SAVANNAH RIVER

Chemical Development
Separations Technology Unit
Technical Section, Engineering Dept.
Hanford Works

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Chief, Declassification Branch CMB

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INTRODUCTION

On January 12, 1953, an explosion occurred in the Savannah River pilot plant which has been used since November, 1951 by du Pont personnel to train operators and to test equipment for their new Purex production plant. The explosion occurred in an evaporator in which a uranyl nitrate solution containing some organics was being concentrated to a high degree to remove nitric acid. This memorandum reviews the facts surrounding the incident as obtained from the official "Preliminary Technical Report",⁽¹⁾ and by talking to du Pont personnel.^(2,3) These facts are interpreted as far as possible and the current design for the Hanford Purex production plant is re-evaluated in the light of these data.

Acknowledgment and thanks are due to the du Pont organization for permission to quote from their preliminary technical report from which much of this memorandum is copied directly.

SUMMARY AND CONCLUSIONS

The summary from the preliminary technical report is quoted below.

"Normal operations which have been carried out in the E-4 evaporator during the past 15 months are:

- (a) Production of process water.
- (b) Concentration of nitric acid.
- (c) Concentration of process feeds containing uranyl nitrate (UNH), nitric acid, and water.

At the time of the explosion an evaporation was in progress for the purpose of decreasing the nitric acid content of approximately 1800 gallons of UNH-HNO₃ solution. Equipment size necessitated that the deacidification be carried out in several batches of approximately 500 gallons each.

Three batches had been successfully processed. The final charge consisted of the 70 gallon heel of the original solution plus 160 gallons of previously evaporated material which had been diluted with water. The additional 160 gallons were required to make a minimum evaporator charge.

The batch was heated with 145 psig steam to the coils until about 1 hour after the specific gravity reached 1.9. The time of the explosion coincided with the completion of the evaporation cycle.

The evaporator was demolished and the plates of the attached column ~~was~~ bowed. The building suffered some structural damage and considerable damage to doors, windows, and siding. All equipment was contaminated with uranium containing dust. Photographs are included which show the extent of damage to the evaporator and building.

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Service piping, other than that immediately associated with the evaporator, was damaged only incidentally.

Most of the instruments, pumps, and other major equipment in the building do not appear to be damaged, but will require an operating check before the total extent of damage to these facilities can be determined.

The evidence has been examined by representatives of the Eastern Laboratory who concluded that a vapor phase deflagration had occurred.

A memorandum summarizing the evidence submitted to Eastern Laboratory and their comments are given in Appendix II.

A search of the literature has shown that several violent decompositions of uranium nitrate solutions have been reported. In each of these cases the indications are that the uranyl nitrate was contaminated with organic.

The finding of organic in the feed and in the evaporator overhead indicates that small quantities of TBP-Amsco were probably in the feed to this charge. Consequently, it now appears probable that this incident was caused by organic contamination. A laboratory study is being formulated to define the conditions under which an explosion will occur so that a recurrence can be avoided. The study will continue of all evidence pertaining to the explosion. The implications of this incident on 221 Building design and operating procedures are also being studied."⁽¹⁾

The tentatively assigned cause of the explosion is a violent vapor phase reaction between nitric acid and butanol.⁽²⁾ It occurred as a solution of uranyl nitrate and nitric acid was being batch concentrated to a high degree (incipient denitration) in the presence of uncertain compounds of partially hydrolysed TBP, uranium, and diluent (known locally as "red oil" and by du Pont personnel as "third phase"). This condition arose as a result of improper organic removal methods and the accumulation of impurities by the recycle of uranium and HNO_3 streams for economic reasons. It is believed that the "red oil" was being rapidly hydrolysed by the high temperature (temperature controller had failed) and acidity existing at the time, resulting in a dangerous concentration of butanol in the vapors above the boiling liquid.

In the current design for the Hanford Purex production plant, the above conditions are not approximated at any point. Uranium nitrate solutions are continuously concentrated at a much lower temperature under conditions conducive to the rapid removal of solvent from the region of boiling liquid. Uranium solutions which will be calcined are steam stripped of solvent prior to concentration. The waste concentration - acid recovery system is also designed to remove the organics continuously to a small residual quantity which is continuously purged with the acid wastes.

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The tanks feeding these concentrators are equipped with skimmers so that any organics which might accumulate for any reason may be skimmed to the utility tank for recovery or disposal. The nitric acid recovered for re-use has been distilled. Organic contamination of this stream is limited to those non-volatile compounds formed by vapor phase reaction during the few seconds it takes for the vapors to pass through the upper portion of the acid fractionator. In view of laboratory data,⁽⁴⁾ this effect is believed to be negligible.

The conditions under which the violent reaction occurred in the Savannah River pilot plant are not approximated in any existing or proposed Hanford facility. In view of this fact, and the successful operation of the Metal Recovery plant and the pilot plants at Hanford, KAPL, and ORNL, no design changes are now planned. Hanford designs will, however, be re-evaluated continually in the light of the experimental data to be obtained at this and other AEC sites. Study is planned to establish the nature and required conditions for reactions of violent nature to demonstrate the magnitude of the margin of safety in the Purex and Metal Recovery production plants.

DISCUSSION

A. Background

For the operation of the Savannah River pilot plant about 2500 pounds of uranium had been obtained from Oak Ridge, and had been used repetitively in the equipment testing program. This uranium had been processed through fifteen Purex IA-IB-IC solvent extractions in addition to various miscellaneous operations such as head end (MnO₂ precipitation) tests, controlled-rate jetting, etc. Re-concentration of the uranium solution was required after each use to return it to a condition simulating process streams. During this time, the aqueous acid wastes from the solvent extraction battery had also been concentrated and re-used in lieu of fresh nitric acid in the preparation of solvent extraction feeds. This concentration and re-use of principal streams prevented all non-volatile organics from being purged from the system via aqueous wastes, and had resulted in the accumulation of several drums of "third phase" which had been skimmed from the process streams. This "third phase", known locally as "red oil", is a mixture of varying composition containing uranium and solvent degradation products such as butyl phosphates, aromatics, organo nitrates, etc.

The concentrator in which all concentrations were performed is described as follows:

"The concentrator (Du Pont drawing W-145175) is 6 ft. in diameter and 5 ft. high on the straight sides. The top and bottom ends are dished. At the top end are nozzles for the column, steam coils, instruments, and inlet and outlet lines. Projecting from the bottom is a 3-ft. diameter by 1 ft.-6 in. deep pot containing the steam coil bundle (1 in. schedule 40 pipe)."⁽¹⁾

The concentrator pot was topped with an 8 plate bubble cap tower. All previous concentrations had been performed with some reflux returned to the top of this tower, but in this particular run, no reflux was used in order that nitric acid might be removed from the boiling UNH solution.

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B. The Incident

The following account is copied from the preliminary technical report.(1)

"To prepare the high-acid UNH solution which resulted from recent mixer-settler studies for the scheduled head-end program, it was necessary (1) to remove ionic impurities, and (2) to deacidify. The ionic impurities, principally sodium, were removed by solvent extraction.

Three methods were considered to remove the excess nitric acid: (1) neutralization with sodium hydroxide, (2) addition of UO_3 , and (3) evaporation. The neutralization with caustic would introduce sodium ions which were undesirable. The addition of UO_3 was the preferred method, but material was not available at the plant site. Therefore, evaporation and removal of nitric acid in the overhead appeared to be the most feasible method.

Deacidification Runs: The first deacidification run was started on December 30, 1952, according to the following procedure: A batch of 500 gallons of UNH solution* was charged to the evaporator, the steam flow was adjusted to 1000 pounds per hour, and maximum water flow was maintained through the condenser.

When the temperature reached 290-300°F, the column was put on total reflux with the steam on to prevent solidification. Water was added, lowering the specific gravity to 1.5-1.6. The charge was cooled to below 150°F, the heel pumped to the hold tank (W-1) and the evaporator flushed with water. Two charges were run successfully in accordance with the above procedure.

The temperature recorder failed prior to the start of the third charge. Based on a comparison of the temperature and specific gravity charts for the two previous charges, it was decided that specific gravity could be used for control by holding the batch with steam on for 1 hour after the specific gravity reached 1.9 (maximum recorder reading 2.0). This procedure was used successfully for the third charge.

The remaining feed material plus water flushes amounted to approximately 70 gallons. In order to complete the evaporation with sufficient heel to cover the steam coils, 160 gallons of material (from W-1) previously deacidified and diluted with water to a specific gravity of 1.6 were added.

* Solution composition was 59% UNH, 3.6% HNO_3 .

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In accordance with the previous procedure, the 230 gallons of solution in the evaporator were heated with a steam flow of 1000 pounds per hour until the start of distillation. At this point, because of the smaller charge, the steam flow was reduced to 500 pounds per hour and held at this rate for the remainder of the distillation.

The specific gravity reached 1.9 at 7:35 P.M. and the evaporation was continued for 1 hour. The explosion occurred at 8:35 P.M. which coincided with the time that the evaporation was scheduled to be completed. The operator had checked the evaporator at 8:28 P.M. and distillate was still coming off. Shortly thereafter a rumbling noise was heard in the evaporator and the operator made the following observation:

'It appeared to me that there were orange colored gases blowing out of the top of the evaporator column with a high-pitched roar. As I reached the laboratory door I observed a large red flash from where I knew the evaporator pot to be. There was considerable concussion at this time.'

Two men were in the laboratory office and four men were in the locker room returning from lunch. Two minor injuries resulted from the explosion.

Facts Noted Relating to Explosion

Analyses and Description of Material Involved

1. The composition of the 70 gallon charge (1.5 sp.gr.) was 47% UNH and 4.3% HNO_3 . This was the tank heel charged from tank E-8 to the evaporator. The composition of this solution did not coincide with the initial feed due to the addition of tank washes.
2. Composition of the 160 gallons (sp.gr. 1.6) was 60% uranium and 1.6% nitric acid. This material, previously deacidified and diluted with water, was charged from tank W-1 to the evaporator E-4.
3. All UNH solutions had been previously contacted with 30% tributyl phosphate in kerosene (Amsco).
4. All UNH solutions had been previously concentrated in the evaporator from sp.gr. of 1.03 to sp.gr. of 1.50 or higher.
5. The distillate from the evaporator was collected in Tank E-8. It contained an organic phase (tentatively identified as Amsco) and an aqueous phase. (See Appendix I, Table I.)
6. Following the explosion, it was determined that W-1 contained an organic phase. (See Appendix I, Table II.) W-1 was used to collect the deacidified solution from the first three charges.

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7. Following the explosion, a greenish yellow solid containing uranium and phosphorous was collected from the immediate environs of the evaporator. (See Appendix I, Table III.)
8. A solid yellow material containing uranium, phosphorous, and NO_3^- was removed from the bottom of the evaporator column. (See Appendix I, Table IV.)

Operating Conditions

1. Steam to coils was 145 psig of saturated steam.
2. Steam flow was at 500 pounds per hour after the start of the distillation.
3. Cooling water was on the condenser at full capacity.
4. Specific gravity indicator read 1.5 sp.gr. at start of run and 1.9 one hour before the explosion.
5. Liquid level indicator showed 230 gallons at start of the evaporation. It had dropped rapidly prior to the explosion and the final reading just prior to the explosion was estimated at 60 gallons.
6. The temperature recorder had failed and was not in operation during this run.
7. The evaporator was vented through an 8-plate bubble cap column to a 3-inch line.
8. Distillate was observed coming from the evaporator at 8:28 P.M. and the explosion occurred at 8:35 P.M.
9. Examination of the evaporator bubble plate column indicated that the charge had probably foamed prior to the explosion.

Survey of Damage

All damage to the building, i.e., girders, beams, footings, etc., was confined to an approximate radius of 12 feet from the evaporator.

The sheet metal on most of the roof and two building sides was either blown off or loosened. The evaporator was demolished and the column plates were bowed."(1)

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Two pages of detailed description of the damage follow in the du Pont report, the description being supported by nine photographs. Additional description of the damage may be found in Appendix II.

C. Interpretation of the Data

The presence of organics in the UNH solution fed to the concentrator is well established, Table I showing essentially Amsco diluent in the condensate and Table II showing "red oil" in the concentrate from the three previous concentrations. Since the final charge to the concentrator included a water flush through the feed tank, it is probable that this final charge contained most of the organic phase which had been floating in the feed tank. Since the charge had been concentrated to about 1/3 its original volume at temperatures from 105 to 125°C with no reflux other than that imposed by heat loss from the equipment, it appears logical to assume that no free Amsco diluent remained in the pot. The presence of Amsco in the condensate does, however, imply that the previous concentrations with reflux had failed to remove all of the Amsco.

The concentration of uranium in the pot at the time of the violent reaction is in doubt. Assuming the estimated volume of 60 gallons contained all of the uranium charged, the specific gravity of the solution would have been about 3.4, a condition not realized until denitration is well under way at temperatures above 160°C. Some of the solution could have foamed into the tower and thus not registered on a liquid level indicator. Assuming the steam rate remained at 500 pounds per hour after the specific gravity reached 1.9, a calculated volume of about 95-100 gallons is obtained for the period just before the blow. If this volume were correct, and the pot temperature about 130°C, the charge would have been concentrated to the point of incipient denitration. Probably some combination of the above possibilities existed.

Under the above conditions, it appears likely that the "red oil" would be hydrolysing rapidly and thus supplying butanol or other volatile organics at an appreciable rate. At the high temperature existing, the HNO_3 or $\text{NO}-\text{NO}_2$ vapors, or both, possibly initiated a vapor phase reaction with the observed results. Experimental work is required to amplify or refute this theory.

D. Future Work

"There is not sufficient information available at the present time to fix definitely the cause of the explosion; however, the presence of organic both in the distillate and the heel is good reason to believe that it may have been involved in the explosion. The accumulation of additional evidence pertaining to the explosion will be continued. An experimental program is also being formulated to define the composition limits within which an explosion will occur so that a recurrence can be avoided.

Concurrent with this work, consideration will be given to the implications which this incident has on the 221 Building design and operating procedures."⁽¹⁾

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Experimental work is also planned by the Hanford Works Technical Section to clarify the problem. This work will attempt to produce a similar vapor phase reaction in the laboratory, then determine the components and temperatures required to produce that reaction. These data will demonstrate the safety of the current design of the Hanford Purex production and the Metal Recovery plants.

E. Application to Hanford Designs

The conditions under which the violent reaction occurred in the Savannah River pilot plant are not approximated in any existing or proposed Hanford facility. In view of this fact, and the successful operation of the Metal Recovery plant and the pilot plants at Hanford, KAPL, and ORNL, no design changes are now planned. Hanford designs will, however, be re-evaluated continually in the light of the experimental data to be obtained at this and other AEC sites.

In the Metal Recovery process as currently being operated, no process stream which has contacted organics is being concentrated in acid media without first having been steam stripped for the removal of volatile organics. No concentrated stream is being returned to process, so that the chances for the accumulation of "red oil" are minimized. The nitric acid which is recovered for reuse is distilled from the uranium stream after the solvent has been removed by steam stripping. Analyses of this recovered nitric acid have revealed a total phosphate content at or lower than the lower limit of detection (< 10 ppm). Since the vapor pressures of all phosphates conceivably present are considerably lower than that of kerosene, it may be concluded that diluent is absent also.

In the Purex design as currently scoped, the uranium product is steam-stripped of solvent prior to concentration to 60% UNH for shipment. A simple equipment change after the plant is built will permit all uranium streams to be steam-stripped prior to concentration if subsequent data indicate its desirability.

The acid wastes are not steam-stripped prior to concentration, and incorporating this step would require a major change of scope. Data⁽⁴⁾ exist, however, which show that greater than 99% of the solvent introduced in the waste concentrators will be removed essentially unchanged with the condensate. That small portion of solvent which may react to form non-volatile products will be purged continuously with the acid wastes and ultimately stored on the alkaline side in the underground waste storage tanks. Contamination of the recovered nitric acid is of necessity limited to that carried by entrainment from the concentrators, or to non-volatile compounds formed by vapor phase reaction during the few seconds the vapors are passing through the acid fractionator.

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APPENDIX I (DPST-53-106)TABLE I

The evaporator distillate in Tank E-8 was analyzed as follows:

<u>Analysis</u>	<u>Organic</u>	<u>Aqueous</u>
Sp.gr. 25°C	.7890	1.0183
Acidity	0.08% HNO ₃	3.0% HNO ₃
*Hydrocarbon	98.4%	
Phosphorous	nil	nil
**KMnO ₄ Test	0.075 ml	
Organic nitrogen	0.28%	nil
Uranium	0.03%	0.12%
Manganese		nil
Butyl alcohol (qual.)	nil	nil
Aniline point (1-1)	63°C (corres- ponds to Amsco 60-65°C)	
Boiling Range ASTM Distillation:		

IBP - 184°C	55 - 196°C
5 - 188°C	65 - 197.5°C
15 - 190°C	75 - 198.0°C
25 - 193°C	85 - 202°C
35 - 194°C	95 - 206°C
45 - 195°C	Dry - Not Done

* By solubility in concentrated H₂SO₄.

** Mls 0.1 N KMnO₄/ml sample.

TABLE II

The organic and aqueous material in Tank W-1 analyzed as follows:

<u>Analysis</u>	<u>Organic</u>	<u>Aqueous</u>
Sp.gr. 25°C	1.47	1.63
Acidity	0.2% HNO ₃	1.5% HNO ₃
*Hydrocarbon	Nil	
Total uranium	26.8%	27.9%
Uranium extractable in .01 N nitric acid	6.2%	
Phosphorous (Total)		.013%
Phosphorous (Inorganic)		.008%
Butyl alcohol (Qual.)	Nil	Nil
NO ₃ ⁻ (Qual.)		Positive
** KMnO ₄ Test		No reducing agents

* By solubility in concentrated H₂SO₄.

** Ml of .1 N KMnO₄/ml of sample.

APPENDIX I (CONT'D.) (DPST-53-106)

TABLE III

Solid Products from Evaporator

A greenish yellow material collected from the immediate vicinity of the exploded evaporator analyzed as follows:

<u>Analysis</u>	<u>Greenish yellow solid contaminated with magnesia insulation</u>
Insoluble in water	77.9%
Insoluble in concentrated HNO ₃ acid	10.6%
105°C volatile	11.5%
Loss on ignition	26.5%
Total uranium	48.8%
Total water soluble	0.5%
Total phosphorous	0.56%
Carbon as CO ₂	Positive
Nitrate (qualitative)	Positive

TABLE IV

A sample of material removed from the bottom of the evaporator column analyzed as follows:

<u>Material</u>	<u>Yellow Solid from Evaporator Column</u>
105°C	10.2%
Loss on ignition	28%
Total uranium	55.2%
Total phosphorous	0.7%
Nitrate (qualitative)	Positive
H ₂ O (hydration)	Not detected
Carbon as CO ₂	Positive

APPENDIX II (DPST-53-106)

REVIEW OF EVAPORATOR EXPLOSION
WITH REPRESENTATIVES OF THE EASTERN LABORATORY

On Monday night, January 12, 1953, at about 8:35 P.M., the evaporator (Equipment Piece 311.15) in the 678-G Building, exploded. The purpose of this report is to comment on the severity of the explosion as indicated by the extent of the damage, and to postulate the type of explosion that took place.

The evaporator (Du Pont drawing W-145175) is 6 ft. in diameter and 5 ft. high on the straight sides. The top and bottom ends are dished: at the top end are nozzles for the column, steam coils, instruments, inlet and outlet lines; on the bottom is located a 3 ft. diameter by 1 ft.-6 in. high pot containing the steam coil bundle (1 in. schedule 40 pipe).

At the time of the explosion a nitric acid solution of UNH was being evaporated, and it is estimated that the volume had been reduced to about 50 gallons, barely enough to cover the coils. According to the recorder charge, the specific gravity had exceeded 1.92 (maximum chart reading) and the instrument had been shut off. The weight factor (liquid level) record is difficult to interpret, but shows a rapid drop down to zero during the 1/2-3/4 hour preceding the explosion. The steam flow record shows an arbitrary reading of about 40 between 6:00 and 6:45 P.M. and then a reduction to 20 where it was held steady until the explosion. Prior to 6:00 P.M. the steam flow had been 0. No temperature or pressure records are available.

Photographs are available which show the extent of the damage to the evaporator, other equipment, and the building. The evaporator was ripped into approximately six pieces. These were all of considerable size, and there appeared to be few, or no small fragments of the missile type. Clean-up operations, already in progress, made a search for missiles unproductive, but there were only three or four small holes in the roof and sides and these might have been made by material other than pieces of the evaporator shell. The top of the evaporator was torn off at a circumference just below the man-hole, on a line corresponding to the ring angle iron to which the four supporting columns were attached. This piece was split up a "meridian" nearly to the column nozzle, and was well opened up. The rest of the evaporator was split into about five pieces of roughly equal size. Two pieces of the 1-1/2 ft. x 3 ft. coil pot were separated, and another piece was still attached to a larger piece of the evaporator bottom by the weld. All of the breaks had the appearance of being ripped; there was no evidence of burning or melting of the metal. The bottom pieces were blackened on the inner surface, but this may have been due to accumulated stains from previous runs.

APPENDIX II (CONT'D) (DPST-53-106)

The coil bundle was squashed slightly, and the outer turns were pushed downward with respect to the inner turns. The draft tube was bent in several places on the bottom edge. These distortions can be explained by a downward force on the coil bundle being resisted by the legs on the draft tube, backed up by the evaporator bottom and drain nozzle against the concrete floor. The bottom nozzle was broken off at the weld. None of the coil pipe (schedule 40 - 1 inch) was collapsed or ruptured.

The column appeared outwardly undamaged, although the bottom tray may be bowed downward slightly. This will be checked. Most of the lines to the column and condenser were still attached. There has been no opportunity to examine the column, condenser, vent, or take-off lines for plugs.

As seen in the photographs, most of the corrugated sheet metal siding was blown off both ends of the building. On the exposed side most of the siding was left intact, but loosened. Only a few roof panels were blown off but most of the roof was loosened. The block partition between the compressor room and the evaporator was demolished to a height of about 10 feet. Damage to adjacent temporary buildings is apparent from the photographs.

The (empty) tank next to the evaporator was dented slightly near the top and moved on its foundation. The rest of the equipment in the building appears relatively undamaged.

The results, as described above, were discussed on an unclassified basis with C. O. Davis and H. H. Fassnacht of Eastern Laboratory, Explosives Department. They agree that probably a low-order vapor phase explosion took place. Davis pointed out that most gas explosions at atmospheric pressure, when oxygen balanced with air, give rise to pressures in the range of 55-160 pounds per square inch. When oxygen balanced with NO_2 , the resulting pressure might be 4 - 5 times this range or enough to rupture the evaporator. The condition of the coil bundle, and mode of rupturing of the evaporator, seems to rule out an explosion in the liquid phase.

Samples of the evaporator feed stock, and condensate are now being analyzed for organic nitrates by Eastern Laboratory. When the results are available, analyses for other components may be requested.

It is planned to make some heat stability tests at Eastern Laboratory in the near future, in which simulated evaporator explosions or reactions will be attempted.

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References

1. DPST 53-106, Preliminary Technical Report TNX Semiworks Incident January 12, 1953, by D. E. Waters, 1-23-53.
2. William Jenkins, du Pont Technical, at Wilmington, De., private conversation, January 22, 1953.
3. D. E. Waters, Chief of Savannah River Pilot Plant, telephone conversation, February 4, 1953.
4. HW-26517, Nitration Reactions of Shell Spray Base Under Purex Waste Recovery Process Conditions, by R. M. Wagner, December 5, 1952.

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