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August 1[illegible], 19[illegible]

1. [illegible]

C[illegible] lead is used in the manufacture of dye intermediates, which [illegible] the backbone of the dye stuffs industry. The performance [illegible] at one plant using lead in [illegible] lead reaction vessels and others, [illegible] agitators, heating and cooling coils and coil [illegible], conveying and drain lines, fume ducts, pumps, valves and other uses [illegible] service life of from 5 to 10 or more years. In many cases original installations made more than 1[illegible] years ago are still performing satisfactorily and undoubtedly will continue to do so.

2. LOCATION

General Aniline & Film Corporation, Grasselli, New Jersey.

3. PROCESS

Dye intermediates are the building stones, so to speak, out of which dyes are constructed with chemical reactions acting as mortar to connect one intermediate with another in manufacturing the dye. The production of intermediates economically and in a pure form is the foundation of the dye industry. Detailed descriptions of various intermediates and their manufacture would be a monumental task as about 700 intermediates, for the most part organic chemicals, are currently being produced. Intermediates manufactured at Grasselli are used not only to make dyes but also may enter into the manufacture of many other products such as explosives, perfumes, medicinals, flavors, resins and plastics. A variety of chemical operations are utilized in the production and processing of intermediates. Briefly these include:

1) Nitration - the introduction of the nitro group (NO_2) into an organic compound by reacting with concentrated nitric acid or a mixture of nitric acid and sulfuric acid [illegible] by removal of water with concentrated sulfuric acid. No lead at this plant - [illegible] used elsewhere.

2) Amination - the introduction of amino groups (NH_2) by reduction with iron and an [illegible] catalyst or by ammonolysis with ammonia. In each case one of the major products is aniline, an important constituent of certain dyes. No lead.

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3) [illegible] - the [illegible] of [illegible] with chlorine or [illegible]. Lead is used [illegible] with cold hydrochloric [illegible].

4) [illegible] - the reaction of organic [illegible] with concentrated sulfuric acid [illegible]. Lead is used afterwards to [illegible] products.

5) [illegible] - the reaction of organic [illegible] with [illegible] or water [illegible]) group [illegible] is [illegible] in this manner. At Grasselli considerable lead equipment is used in this process.

6) <u>Oxidation</u> - simply controlled combustion which may involve burning in air or [illegible] with oxidizing chemicals to introduce the (O) radical. Lead is used extensively at Grasselli.

7) <u>Alkylation</u> - the introduction of an alkyl radical [$(CH_3)_2$] by substitution or addition into an organic compound with the aid of sulfuric acid. Lead is not used in actual process but later on is used to handle products of reaction.

8) <u>Esterification</u> - in the presence of some sulfuric acid, acids and alcohols are converted to esters such as nitroglycerine or nitrocellulose. Lead equipment is used to a great extent.

9) <u>Reduction</u> - chemical change involving a loss of oxygen or gain of hydrogen. At Grasselli homogenized lead equipment serves exclusively.

10) <u>Extraction</u> - a method by which soluble parts are separated from a substance. This is usually accomplished by heating in jacketed vessels or by means of coils. At Grasselli lead coils are used for heating and later condensing. Often jacketed homogenized lead vessels are used.

11) <u>Condensation</u> - most often a change from vapor to liquid state by cooling but sometimes a chemical reaction involving combination of molecules with separation of water. Homogeneous lead bonded vessels are used in this process at Grasselli.

At Grasselli the above processes are used at one time or another in the manufacture of their many products of which dye intermediates constitute the major part. As indicated, much of the equipment used in these processes consists of lead as vessel linings (primarily homogeneous), heat exchanger coils, conveying lines and valves, protective covering for agitator blades, pumps, fume ducts, and many other miscellaneous uses typically encountered whenever corrosives are handled. In some of the above processes concentrated sulfuric acid (above 96 per cent) is used during the reaction. In that case no lead is utilized; however, after the reaction is completed, the acidified product must be diluted and occasionally neutralized. Here lead equipment serves exclusively.

4. CONSTRUCTION DETAILS

A few of the many interesting lead installations at the Grasselli Works of the General Aniline & Film Corporation were examined in detail recently and are described below. In all cases lead was substituted along with other materials to comprehensive laboratory tests under conditions approaching as closely as possible those found in actual service before selecting it for installation. Selection of materials of construction for intermediate manufacture is complicated to the extent that side reactions often result in products which attack linings ordinarily resistant to the major corrosive constituents. For this reason extensive tests must be conducted before final material selection so that the material offering the most universal protection is chosen for the job.

See accompanying drawings for construction details of the equipment described below.

A. Reaction Vessels

The reaction vessels average 12 to 18 ft. in depth and 6 to 8 ft. in diameter. Most of them consist of a steel shell double lined with acid-resistant brick and no lead lining. Some of the jacketed vessels, however, are homogeneously lead bonded with no brick lining. Lead heating and cooling coils, lead coil supports, homogeneously bonded lead agitators and bonded lead vessel covers are utilized in the brick lined vessels. One of that type examined handled 5 per cent sodium bichromate plus 20 per cent sulfuric acid at temperatures ranging from 200° to 240° F. No evidence of lead corrosion was apparent and no trouble had been encountered from this cause.

The use of lead in these vessels is particularly noteworthy inasmuch as the presence of even minute quantities of metallic salts would seriously affect the quality of the finished product. Lead's reaction with the corrosives handled does not result in the formation of any corrosion products which might hinder in any degree the success of the operation. The covers on these vessels which are often operated under pressures other than atmospheric are constructed of steel homogeneously bonded with lead from 1/4 in. to 5/16 in. thick. Tanks are emptied by blowing contents through a lead pipe extending through the top to the bottom.

B. Coils

Each vessel not heated externally is equipped with an internal lead heating and cooling coil constructed of tellurium lead pipe 2 in. I.D. and 3/8 in. wall thickness. The coils handle 30-lb. steam pressure for heating and cold water for cooling. They have given excellent service, the only repairs necessary being the correction of slight sagging on some of the higher temperature reaction vessels.

C. Coil Support

Of particular interest in these installations is the design of the all-lead coil support consisting of chemical lead cast in bars 3 x 1½ in. in cross-section. Two bars extend up the inside of the tank where the coil is fastened to the support by means of welded lead straps. All supporting bars converge at the hemispherical base of the tank. This method was developed at Grasselli as a replacement for bonded lead steel supports. According to R. C. Toledo, plant engineer, this method of construction "...permits uniform bonding between coil and supports; it facilitates construction and assembly; eliminates the possibility of iron contamination deleterious to dye stuffs manufacture and is easily maintained." This type of structure is necessary since the coil cannot be hung from the tank wall without penetrating the acid-brick lining.

D. Agitators

Agitator blades suspended centrally from above through a stuffing box are used to keep the chemicals in motion during the reaction. The steel blades for this purpose are homogeneously bonded with lead 1/4 in. thick. The shafts of the agitators on the other hand, are simply covered with sheet lead welded together. Experience showed that stresses set up in the shaft of the agitator tended to rupture the lead when it was homogeneously bonded. Since loose lead covered shafts were put into service no trouble has been encountered on this score.

E. Fume Ducts

During the reaction of the various chemicals in these vessels, corrosive fumes are given off. Different reactions in each vessel result in a variety of corrosive fumes which include sulfur dioxide, sulfur trioxide, chlorine, bromine and nitrous oxide. Any one of these gases particularly in combination with the air and moisture normally present in the system can constitute a serious corrosion problem. The only material that has been able to withstand successfully the corrosive attack of all of these gases combined is lead. All reaction gases are exhausted from the individual vessels, collected in 10 and 12 in. diameter lead fume exhaust ducts and carried to a 16 in. diameter lead duct from whence they pass into fume removal scrubbers. All of these lines which consist of chemical lead with a 3/8 in. wall thickness have been in continuous service for about 12 years with little or no repairs - a remarkable record considering the nature of the corrosives. Instead of valves, lead dampers are used for by-pass arrangements. All fume ducts were fabricated on the plant premises from 20-lb. chemical sheet lead and are strapped to steel channels which provide a supporting framework. Horizontal sections are strapped on the upper halves with steel and covered with lead, the undersides resting on steel channels. Smaller diameter lines are simply laid in steel channels hung from above.

F. Other Lead Equipment

Lead drain lines and lead floor drains are used for handling waste acids and waste dye intermediates. In most instances tellurium lead piping with 1/2 in. wall thickness is used. Lead-lined valves are used throughout.

One of the constituents of dye intermediate processing is trinitrotoluene (TNT). Because of the explosive nature of this material, the steel shovels used for emptying product trucks would constitute a dangerous hazard if the nonsparking characteristics of lead were not used to advantage. All of the steel product trucks used for handling corrosive acid filter press cake are Loco-lead lined with 8-lbs., 6 per cent antimonial sheet lead. The trucks are 2 x 4 ft. on top and 2 ft. deep.

5. PERFORMANCE

All vessels and vessel covers which were homogeneously bonded with lead from 1/4 in. to 5/16 in. thick have been performing successfully since their installation, which averages between 5 and 10 years ago. The same excellent service has also been given by lead coils and supports and bonded lead agitators - the only deviation being provided by the latter which performed better after the shafts were sheet lead covered instead of lead bonded.

Fume ducts gave excellent service since their erection 12 years ago and will undoubtedly continue to resist successfully corrosive fume attack. The efficient manner by which they are supported contributed in large measure to the longevity of these ducts (see drawings). Waste acid drain lines also have served for 12 years, as well as a quantity of lead-lined valves and other miscellaneous lead equipment.

6. CONCLUSION

An intricate science, the manufacture of intermediates incorporates many combinations of a variety of corrosive chemicals under a variety of conditions of concentration, temperature and agitation. Because lead is resistant to many more corrosives and combinations of corrosives than any other comparable corrosives handling material, it successfully protects much of the process equipment in this important industry.

All of the lead work at Grasselli is done in a well-equipped shop located on the premises with the exception of some major construction let out to contractors. A result of many years' experience with lead equipment (dating back to the days of the first World War when German-made dyes were shut off from the Allies) GAF engineers have learned that lead, properly applied and treated with due respect, will serve most economically. To this end they have specified homogeneous bonding wherever possible and apply the lead generously, at least 1/4 in. thick and oftentimes 3/4 in. thick. By adopting the philosophy of spending a little more initially instead of a lot later on for repairs, GAF has reaped a reward of excellent service from its lead equipment. Service life for all of the lead installations described above has ranged from 5 to 10 or more years and in many cases original equipment is still performing satisfactorily after more than 12 years of use. The majority of all lead equipment at this plant has more than paid for its original installation costs and, moreover, has not been replaced at any time by any substitute material.

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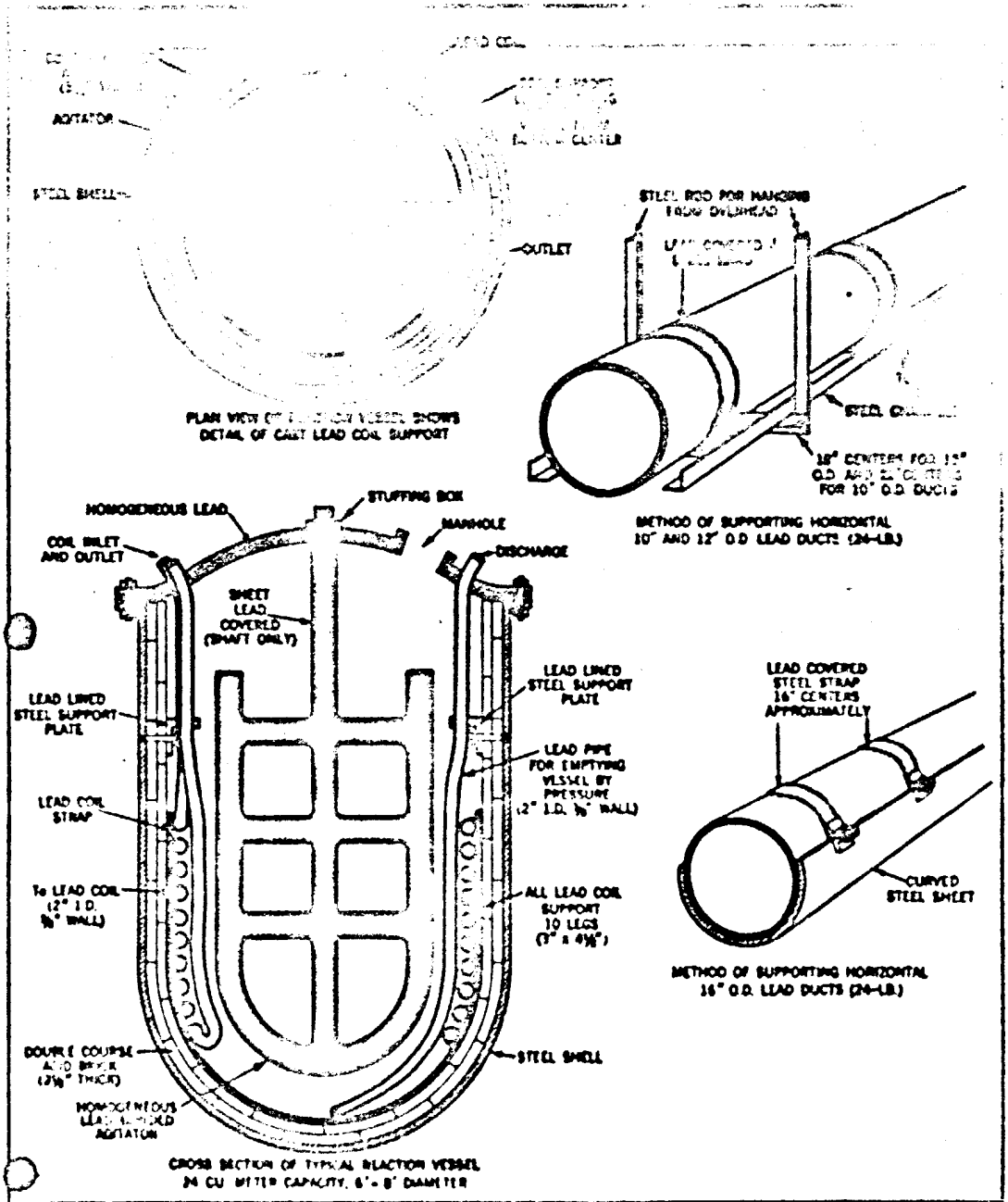

REACTION VESSEL AND FUME DUCT SUPPORTS BY R. H. ROLL AUGUST, 1949	LEAD INDUSTRIES ASSOCIATION 420 LEXINGTON AVE. N.Y. 17, N.Y.	BRASSELL WORKS GENERAL ANILINE AND FILM CORP. CHEMICAL BULLETIN NO. 58