

## ON THE ORIGINS OF A TOOL FOR CHEMISTS, THE DEAN-STARK APPARATUS

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### Summary

An utterly simple glassware tool for chemists, the Dean-Stark trap as it became known, was devised in 1920. This paper strives to elicit the multiple meanings carried by this little device, with widespread and continued use in the chemical laboratory. It also describes the milieu of American chemistry between the two World Wars in which the original paper by Drs. Dean and Stark appeared.

### Introduction

During most of the twentieth century, chemistry laboratories, whether academic or industrial, contained separate enclosures: the processed material had to be protected from two main enemies, dioxygen and moisture. Hence, many shielding devices were resorted to: for the former, running reactions and separations under helium, argon or dinitrogen, and vacuum lines. For the latter, laboratories displayed ovens, conservation of solvents over sodium wire, desiccants such as Drierite® or the Fisher 4-Å molecular sieve, desiccators, dry boxes, and more.

We shall concern ourselves here with yet another laboratory tool against moisture and for its removal, the Dean-Stark apparatus (also known as the Dean-Stark trap) (1). We shall focus primarily on the early 1920s when this device was invented. Such a viewpoint, making explicit the invisible birthmarks on an artefact, is

comparable to establishing the etymology of a word. Another, near orthogonal take, is comparable, in turn, to chronicle the evolution in the uses and meanings of this word, across the ages, as Jean Starobinski beautifully did for the word “reaction” (2). Both approaches, we submit, are valid historical contributions.

### Birthplace

This tool for chemists was devised in 1920 at the Pittsburgh Station of the US Bureau of Mines. Its progenitors were two scientists from the chemistry section, Edward Woodward Dean (1888-1959), a Yale Ph. D. in 1912 with W. A. Druschel, and David Dewey Stark, a junior petroleum chemist (1893-1979) (3). The actual maker was the glassblower for the Station, Francis E. Donath (1874-ca. 1960).

The US Bureau of Mines concerned itself with chemistry, and with the chemistry of petroleum in this case, as a not-too-distant consequence of the breakup, in 1911, of the monopoly of Standard Oil and the so-called “independents.” At the outbreak of World War I, the Federal administration and the newly-formed oil companies decided to work together, rather than in opposition, to ensure continued supply of oil-derived fuel to the military. On March 20, 1919, the American Petroleum Institute was started in New York. Its missions included collection of statistics for the oil industry and development of industry-wide standards (4).

This was indeed a period for establishment of public petroleum institutes in various countries. For example, in France, at the newly regained University of Strasbourg—the Alsace-Lorraine was returned to France from the German occupation it had undergone since 1871, after having been part of France since the seventeenth century—Henry Gault (1880-1967) a professor of chemistry, founded in 1920 the *Institut du pétrole et des moteurs*. It served as the seed for what would later become, when it departed Strasbourg after World War II, *Institut français du pétrole*.

Let us now return to the Pittsburgh Station of the US Bureau of Mines. Pittsburgh then numbered 600,000 people and it was the fifth largest metropolis in the United States (5). Fossil fuels, coal and petroleum had made it a thriving industrial center (6). The former, an abundant local mining resource, had given rise to metallurgy in the area. Pittsburgh is at the center of the northern half of the great Appalachian coal field. Beneath the so-called “Pittsburgh seam” there was another, the Freeport seam, which ensured durable coal production. In the 1920s annual coal production was in the order of 100 Mt.

As for petroleum, one will recall, underground deposits had been discovered by Titus Drake in Pennsylvania. As a consequence, Pittsburgh hosted oil refineries, no fewer than 58 in 1867 and at that time supplied over 60 % of the entire foreign export of petroleum (7). Pittsburgh in the early 1920s was a techno-city (8) that included a center of chemical research (9).

Coal, steel and oil made hefty contributions to the fortunes of the Carnegie and Mellon (10) families. As John Dos Passos wrote in *The 42nd Parallel*, published in 1930 (11),

Andrew Carnegie believed in iron, built bridges Bessemer plants blast furnaces rolling mills;

Andrew Carnegie believed in oil;

Andrew Carnegie believed in steel;

always saved his money

whenever he had a million dollars he invested it.

Andrew Carnegie became the richest man in the world and died.

Bessemer Duquesne Rankin Pittsburgh Bethlehem Gary ...

A new building of the US Bureau of Mines was dedicated in 1919—construction had begun in 1915 (12)

and actual occupation and use started in 1917—on Forbes Avenue in the Squirrel Hill neighborhood (13). It was a neighbor to another two laboratories also devoted largely to applied science, the Carnegie Institute of Technology, founded in 1900, and the Mellon Institute of Industrial Research, founded in 1913 by Andrew W. Mellon.

The research staff of the US Bureau of Mines in their new building immediately began working on the physico-chemical properties of crude oil, such as viscosities, from various production sites in the US (14). Edward W. Dean worked there from the opening of the Station. He studied at first petroleum distillation and gasoline manufacture (15). Dr. Dean was responsible for reports on the properties of American crude oil from various locations (16). He took responsibility also for the design of laboratory instrumentation (17).

### Hydrocarbon Fuels During and After World War I

In the aftermath of the Great War, two modes of transportation, aviation and the automobile, drew heavily on production by oil refineries in the United States. The Twenties roared also with automobile and airplane engines. The former is illustrated by the biography of Horatio Alger’s father, an aviation pioneer in the Army Air Corps who entered the reserves in the late 1920s and then became an aviation fuel expert for Standard Oil Company. Alcock and Brown used Shell fuel to make the first trans-Atlantic flight in 1919.

The 1920s were a period of building American airports. In Chicago, for instance, originally built in 1923 as the Chicago Air Park, Midway airport was mainly used initially by airmail contractors. In 1927 it was dedicated as the Chicago Municipal Airport. 1928 marked the airport’s first full year of operation with 12 hangars and four lighted runways to allow night flights.

Following Charles Lindbergh’s solo and non-stop transatlantic crossing (1927), airlines began sprouting in various countries. In Europe, the French Aéropostale had opened its first route between Toulouse and Barcelona just after the end of the Great War. It was extended to Casablanca by February 1919, to Dakar by 1925, and to South America by the spring of 1930. Duration of intercontinental travel was cut down from days to hours.

As for the automobile, before the Great War, cars were a luxury reserved to the very wealthy. In the 1920s, mass-produced vehicles became common throughout North America (18). By 1927, Ford discontinued the

Model T after selling 15 million of them. Gasoline, although differing from aviation fuel, also had as a requirement the presence of an anti-knock agent, in the form of tetraethyllead: this innovation, together with the devising of the octane rating (19), also dates to the same period of the 1920s.



**Figure 1.** A Penn Oil Truck from 1920 boasts “more power” (Library of Congress Prints and Photographs Division, National Photo Company Collection).

Hence, oil refineries in the 1920s had to supply standardized fuels with well-defined characteristics (octane 40-60), in the face of crude oil beset with highly variable parameters, not to mention its admixture with water (or rather brine) as it came out of a well—which was the origin of Dean and Stark’s search for an efficient separation procedure or device.

### The Devising of the Tool

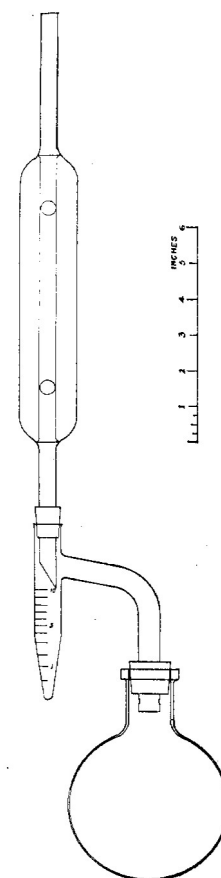
In the immediate aftermath to World War I, when Dean and Stark announced their device, the presence of moisture in various commodities, such as indeed petroleum, but also quite a few others, was a nagging technological concern: how to measure its level, how to get rid of it. The same *Industrial and Engineering Chemistry*, during the year 1920 when Dean and Stark’s paper appeared, carried others on that very topic (20).

The scientists, physicists and chemists, involved in petroleum studies faced the frequent occurrence of water-in-oil emulsions, which led to samples in flasks and test tubes “frothing at the mouth” so to speak. This was a problem in the field as well (21).

The two progenitors, Drs. Dean and Stark, conceived a solution. It combined the can-do mentality and the American genius for putting together a device that will do the job. Their Rube Goldberg device was, to put it in the simplest possible manner, a hybrid between a still and a funnel.

The technical problem, once analyzed, was how to separate water from a hydrocarbon solvent. The technical fix was obvious, a distillation together with removal of the offending water. Addition of a hydrocarbon solvent would make sure the mixture was removed from the azeotrope. After experimenting with a number of adjunct solvents, Dean and Stark elected either a petroleum naphtha (“cleaner’s naphtha, of proper distilling range”) or a benzene-xylene mixture. Water had the greater density, hence gravity would suffice to remove it. It was only necessary to put in an adjunct to the condenser, in the form of a small separate container for the water. The glassblower at the Station, Mr. Donath, made the all-glass contraption, henceforth to be known as the Dean-Stark adapter, apparatus, or trap. Here is their diagram of the device (Figure 2):

Drs. Dean and Stark chose to publish in *Industrial and Engineering Chemistry*. It was an outstanding choice. This periodical of the American Chemical Society was obviously devoted to applied science. Still, it emphasized a tight coupling with pure science as shown, for instance, by the publication the same year 1920 as Dean and Stark’s report of an article by Irving Langmuir on the novel understanding of the chemical bond gained in the aftermath of G. N. Lewis’s 1916 paper in *JACS* (22). It published articles on chemical education and the training of chemical engineers (23). Another sign of the breadth of the interests represented by that journal was its concern with economic



**Figure 2.** The original apparatus, as depicted in Dean and Stark’s paper (Ref. 3).

geography: each issue carried reports on the chemical industry overseas or in various parts of the United States. In terms of technical problems, crude oil clearly was a major concern, as other articles published the same year 1920 attest (24). In short, a chemical engineer and many a research chemist would have been well inspired to read each monthly issue from cover to cover.

What about the writing of Dean and Stark's paper? Two qualities strike today's reader, nearly a century later. The first feature is the total absence of hype. To quote these authors, "On account of the simplicity of the modified method the authors have found it difficult to believe that their work could be new." The second feature is the detailed, exhaustive, step-by-step description of both the distillation procedure and the apparatus: this was a time when scientific reproducibility was clearly at a premium, the editors would not have tolerated any but a report which anyone could reproduce easily.

### **Pittsburgh Recession**

About that time in the early 1920s, Pittsburgh entered a decline in attractiveness to industry and thus lost its prosperity. The City Fathers made an effort at conversion from leadership in industry to another in scientific research and in education (6).

As a memory of its heyday as a world center in oil refining, major oil companies made their headquarters in Pittsburgh. For instance, in 1922, the Penn-Okla Oil Corporation had its headquarters in Pittsburgh. Of course, Pittsburgh prided itself on the Mellon family and its fabulous wealth, which had accrued significantly from oil production and distribution. Accordingly, the headquarters for both Mellon properties, Mellon Financial and Gulf Oil, were and stayed in Pittsburgh. The most impressive Gulf Tower skyscraper was completed in 1932. In addition, Gulf Oil maintained its research and development laboratories in Pittsburgh, until its takeover by Chevron. The oil legacy was thus still alive in Pittsburgh past the 1950s (25).

The Cathedral of Learning tower, commissioned in 1921, went up in 1931-34 and it signaled the ambitions of the University of Pittsburgh. The nearby institutes for applied science, whether the Carnegie, the Mellon or the US Bureau of Mines forged on. Their part of the city underwent continued development until the Depression. To mention just one landmark from that time, the Webster Hall hotel was built in 1925. It remains to this day a monument to Pittsburgh's Golden era, that lasted

for half-a-century, from the 1870s until the 1920s.

What about our three inventors, Drs. Dean and Stark, and Mr. Demuth? The first two, alert and smart scientists that they were, saw the writing on the wall. Oil prospecting and production were leaving Pennsylvania behind, electing to settle instead in places such as Oklahoma, Louisiana and Texas, and overseas locations such as the Persian Gulf. Of those two scientists, one moved East, the other moved West: in 1922 Dean moved to Standard Oil's labs in New Jersey (26) and Stark went to the Bay Area, where he worked for Associated Oil in San Francisco (27). As for Mr. Demuth, he stayed put, continuing in his work as a glassblower in the Station. In fact, he would retire in 1952, after 40 years of service to the US Bureau of Mines in its Pittsburgh Station (28).

### **Conclusion: the Coming of Age of American Chemistry**

The devising of the Dean-Stark apparatus is not an isolated event, rather it is part and parcel of the impressive rise of American science, chemistry in particular, following the end of the First World War. The same year 1920 as Dean and Stark's publication, George Eastman started production of laboratory chemicals in Tennessee (29). Laboratory glassware started to carry a distinctly American trademark with the production of pyrex glass by the Corning company (30). Later developments, ushering in a revolution in the tools of chemistry completing the switch from a craft to a profession, if they came later, during the 1930s, were also American: the building of X-ray diffractometers, of electron diffractometers, of mass spectrometers (with their first appearance within petroleum chemistry as well), of infrared spectrometers (31), of the microelectrode by Ida H. Hyde (1857-1945) (32), ... All were signs of the times. Among other laboratory tools devised by Americans between the two world wars, pride of place might go to the pH meter, invented around 1936 by Arnold Beckman. A trickle-down from academia (Caltech), it shared with the Dean-Stark trap being a device for solving practical problems of field work (33).

Let me briefly list some of the factors for the rise of American chemistry to the fore: the boost of the economy due to the war in Europe (34); the World War I defeat of Germany, of course, and the new role of the US as the world economic leader; the passing of the baton from Germany to the US in dyes manufacturing (35); other technological transfers (36); electrification of the country (37); industrialization of the West Coast (38) and the



setting-up on its campuses of large chemistry institutes on the German model (whereas on the East Coast, at least in the Ivy League, the British tradition continued to rule); somewhat earlier on, during the period prior to the Great War, the start in the US of professional institutions of lasting value such as, to mention but a few, in addition to *Industrial and Engineering Chemistry*, the Chemical Abstracts Service (1907), annual meetings of the American Institute of Chemical Engineers (1908); enrollment of women among engineers and scientists (39); and last but not least, great American scientists (40). Other assets, belonging to mentalities, were more than a taste: a yearning and a need for innovation; a well-educated elite; the size and diversity of the country; and, last but not least, sharing with the British not only their language, also an ambition of scientific excellence.

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